PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 4 : C03C 13/00, 13/02, 25/06

A2

(11) International Publication Number: WO 89/12032

(43) International Publication Date: 14 December 1989 (14.12.89)

(21) International Application Number: PCT/US89/02288

(22) International Filing Date: 25 May 1989 (25.05.89)

(30) Priority data:
201,513 1 June 1988 (01.06.88) US

(71) Applicant: MANVILLE SALES CORPORATION [US/US]; Manville Plaza, 5th Floor, P.O. Box 5108, Denver, CO 80217 (US).

(72) Inventors: OLDS, Leonard, Elmo ; 977 South Lake Gulch Road, Castle Rock, CO 80104 (US). KIELMEYER, William, Henry ; 3374 West Chenango Avenue, Englewood, CO 80110 (US).

(74) Agent: SCHRAMM, William, J.; Brooks & Kushman, 2000 Town Center, Suite 2000, Southfield, MI 48075 (US).

(81) Designated States: AT (European patent), AU, BE (European patent), BR, CH (European patent), DE (European patent), DK, FI, FR (European patent), GB (European patent), IT (European patent), JP, KP, KR, LU (European patent), NL (European patent), NO, SE (European patent).

**Published**
*Without international search report and to be republished upon receipt of that report.*

(54) Title: PROCESS FOR DECOMPOSING AN INORGANIC FIBER

(57) Abstract

Inorganic fibers which have a silicon extraction of greater than 0.02 wt% Si/day in physiological saline solutions. The fiber contains $SiO_2$, MgO, CaO, and at least one of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof. Also disclosed are inorganic fibers which have diameters of less than 3.5 microns and which pass the ASTM E-119 two hour fire test when processed into a fiber blanket having a bulk density in the range of about 1.5 to 3 pcf.

*FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

| | | | | | |
|---|---|---|---|---|---|
| AT | Austria | FI | Finland | ML | Mali |
| AU | Australia | FR | France | MR | Mauritania |
| BB | Barbados | GA | Gabon | MW | Malawi |
| BE | Belgium | GB | United Kingdom | NL | Netherlands |
| BF | Burkina Fasso | HU | Hungary | NO | Norway |
| BG | Bulgaria | IT | Italy | RO | Romania |
| BJ | Benin | JP | Japan | SD | Sudan |
| BR | Brazil | KP | Democratic People's Republic of Korea | SE | Sweden |
| CF | Central African Republic | KR | Republic of Korea | SN | Senegal |
| CG | Congo | LI | Liechtenstein | SU | Soviet Union |
| CH | Switzerland | LK | Sri Lanka | TD | Chad |
| CM | Cameroon | LU | Luxembourg | TG | Togo |
| DE | Germany, Federal Republic of | MC | Monaco | US | United States of America |
| DK | Denmark | MG | Madagascar | | |
| ES | Spain | | | | |

# PROCESS FOR DECOMPOSING AN INORGANIC FIBER

## FIELD OF INVENTION

This invention relates to inorganic fiber compositions and more particularly it relates to inorganic fiber compositions which can contain silica, magnesia, calcium oxide, alumina, and other oxides. Some of the inventive fibers have excellent fire ratings, some have especially low durabilities in physiological saline solutions, and some have combinations of these foregoing properties.

## BACKGROUND OF THE INVENTION

For many years, inorganic fibers generically referred to in the industry as "mineral wool fibers", made from slag, rock, fly ash, and other by-product raw materials have been manufactured. These fibers have been typically manufactured by melting the slag, rock, etc., containing such oxides as silica, alumina, iron oxide (ferrous and ferric), calcium oxide, and magnesia; allowing the molten material to be blown by gas or steam or to impinge on rotors at high speeds; and causing the resulting blown or spun fibers to be accumulated on a collecting surface. These fibers are then used in bulk or in the form of mates, blankets, and the like as both low and high temperature insulation. U.S. Patent No. 2,576,312 discloses a conventional mineral wool composition and method for making the same.

In the past, the industry has well recognized the standard drawbacks associated with conventional mineral wool fibers. Conventional mineral wool fibers may have high contents of undesired oxides which often

detract from their refractory properties. The conventional mineral wools are coarse, i.e. they have average fiber diameters of 4 to 5 microns (measured microscopically) and have high shot contents in the range of 30 to 50 weight percent. The coarseness of the fiber reduces the insulating value of the fiber and makes conventional mineral wool unpleasant to handle and unfriendly to the touch. For example, because of their coarse fiber diameters, conventional mineral wool blankets must have bulk densities of from 4 to 8 pcf and even higher in order to pass the ASTM E-119 two hour fire test. On the other hand, fiber glass blankets are often made with bulk densities of 2 pcf or lower. While the fiber glass blankets are friendly because of their low bulk densities and relatively fine fiber diameter, they do not have sufficient fire resistance so as to pass even the one hour ASTM E-119 fire test.

Recently, another potential problem with traditional mineral wool and other types of fiber has been recognized. It is well known that inhalation of certain types of fiber can lead to elevated incidence of respiratory disease, including cancers of the lung and surrounding body tissue. Several occurrences are well-documented in humans for several types of asbestos fiber. Although for other varieties of natural and manmade mineral fiber direct and unequivocal evidence for respiratory disease is lacking, the potential for such occurrence has been inferred from results of tests on laboratory animals. In the absence or insufficiency of direct human epidemiological data, results from fiber inhalation or implantation studies on animals provides the best "baseline information" from which to extrapolate disease potential.

Chronic toxicological studies on animals have, however, been able to statistically demonstrate the importance of three key factors that relate directly to the potential for respiratory disease and especially carcinoma: (a) dose of fiber received (including time of exposure); (b) dimension of the inhaled fiber; and (c) persistence of the fiber within the lung. The effects of dose and dimension have been well-characterized from such studies and as a result are fairly well known in regard to human disease potential. The dose is obviously a product of the environment in which the fiber is used and the manner in which it is used. The dimension and persistence of the fiber within the lung, on the other hand, are functions of the manner in which the fiber is formed and of its chemical composition. In general, the smaller the fiber the more likely that it will become embedded in lung tissue when inhaled, thus increasing the danger of respiratory disease.

Although less is known about the link between persistence of the fiber within the lung and respiratory disease, increasing attention is being focused on this aspect of the health issue. Biological persistence refers to the length of time a fiber endures as an entity within the body. The physiochemical concept that most closely relates to persistence and is perhaps more easily quantified is that of "durability" - specifically, the chemical solubility (or resistance to solubility) of fibers in body fluids and the tendency of such fibers to maintain physical integrity within such an environment. In general, the less durable a fiber is, the less will be the potential health risk associated with the inhalation of that fiber. One method of measuring the chemical durability of a fiber in body fluids is to measure its durability in physiological

saline solutions. This can be done by quantifying the rate of extraction of a chemical component of the fiber such as silicon into the physiological saline solution over a certain period of time.

Thus, as can be easily concluded from the foregoing discussion, conventional mineral wool fibers have several serious drawbacks. However, even the alternatives to mineral wools have problems. For example, as mentioned earlier glass fibers have a fire resistance problem and whereas the refractory ceramic fibers have been gaining increasing use in recent years as an alternative to mineral wool fibers because of their ultra-high temperature resistance and superior ability to pass all fire rating tests, their use is limited by the fact that they are relatively expensive and have a relatively high chemical durability in physiological saline solutions as well.

In conclusion, there is a great need in the industry for low cost, friendly feeling low bulk density inorganic fibers which have good fire resistance properties as measured by their ability to pass the ASTM E-119 two hour fire test. Additionally, there is a tremendous demand for fibers which have especially low durabilities in physiological saline solutions. What would be particularly advantageous to the industry would be fibers with combinations of the above mentioned sought after properties. Also, advantageous would be fibers which also have excellent refractory properties as well, e.g. high continuous service temperatures.

## SUMMARY OF THE INVENTION

In one embodiment of the present invention, there are provided inorganic fibers having a silicon extraction of greater than about 0.02 wt% Si/day in physiological saline solutions and a composition consisting essentially of about 0-10 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 35-70 wt% $SiO_2$; 0-50 wt% MgO; and CaO.

In another embodiment of the present invention, there are provided inorganic fibers which have a 5 hour silicon extraction in physiological saline solutions of at least about 10 ppm. These fibers can broadly have compositions consisting essentially of the following ingredients at the indicated weight percentage levels:

0-1.5 wt% of either $Al_3O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 40-70 wt% $SiO_2$; 0-50 wt% MgO; and CaO

1.5-3 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 40-66 wt% $SiO_2$; 0-50 wt% MgO; and CaO

3-4 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 40-64 wt% $SiO_2$; 0-50 wt% MgO; and CaO

4-6 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 40-59 wt% $SiO_2$; 0-25 wt% MgO; and CaO

6-8 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 35-54 wt% $SiO_2$; 0-25 wt% MgO; and CaO

8-10 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 35-45 wt% $SiO_2$; 0-20 wt% MgO; and CaO

In a preferred embodiment, inventive fibers with 5 hour silicon extractions of greater than about 20 ppm and most preferably greater than about 50 ppm are provided.

In another embodiment of the present invention there are provided inorganic fibers having a diameter of less than 3.5 microns and which pass the ASTM E-119 two hour fire test when processed into a fiber blanket having a bulk density in the range of about 1.5 to 3 pcf and having a composition consisting essentially of about: 0-10 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 58-70 wt% $SiO_2$; 0-21 wt% MgO; 0-2 wt% alkali metal oxides; and CaO and wherein the amount of alumina + zirconia is less than 6 wt% and the amount of iron oxides or alumina + iron oxides is less than 2 wt%. Preferably, the inventive fibers in this embodiment may have compositions consisting essentially of about:

0-1.5 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 58.5-70 wt% $SiO_2$; 0-21 wt% MgO; 0-2 wt% alkali metal oxides; and CaO

greater than 1.5 wt% up to and including 3 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 58.5-66 wt% $SiO_2$; 0-21 wt% MgO; 0-2 wt% alkali metal oxides; and CaO

greater than 3 wt% up to and including 4 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 58-63 wt% $SiO_2$; 0-8 wt% MgO; 0-2 wt% alkali metal oxides; and CaO

greater than 4 wt% up to and including 6 wt% of either $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof; 58-59 wt% $SiO_2$; 0-7 wt% MgO; 0-2% alkali metal oxides; and CaO.

As discussed herein earlier, there has been a demand in the industry for inorganic fibers with an excellent fire rating at low bulk densities and fibers with especially low chemical durabilities in physiological saline solutions. Therefore, each category of inventive fibers should fulfill a real need in the industry and should be available for applications where heretofore low cost, mineral wool type fibers have not been available. What is particularly advantageous about the present invention is the fact that fibers are provided where a special demand exists, i.e. applications in the industry where fibers with both an excellent fire rating and an especially low durability in physiological saline solutions are in demand.

Other features and aspects, as well as the various benefits and advantages, of the present invention will be made clear in the more detailed description which follows.

## DETAILED DESCRIPTION OF THE INVENTION

The inventive fiber compositions of the present invention can be made from either pure metal oxides or less pure raw materials which contain the desired metal oxides. Table 1 herein gives an analysis of some of the various raw materials which can be used to make inventive fiber compositions. Physical variables of the raw materials such as particle size may be chosen on the basis of cost, handleability, and similar considerations.

Except for melting, the inventive fibers are formed in conventional inorganic fiber forming equipment

and by using standard inorganic fiber forming techniques as known to those skilled in the art. Preferably, production will entail electric furnace melting rather than cupola melting since electric melting keeps molten oxides of either pure or less pure raw materials more fully oxidized thereby producing longer fibers and stronger products. The various pure oxides or less pure raw materials are granulated to a size commonly used for electric melting or they may be purchased already so granulated.

The granulated raw materials are then mixed together and fed to an electric furnace where they are melted by electric resistance melting with electrodes preferably positioned according to the teachings of U.S. Patent No. 4,351,054. Melt formation can be either continuous or batchwise although the former is preferred. The molten mixture of oxides is then fiberized as disclosed in U.S. Patent No. 4,238,213.

While the fiberization techniques taught in U.S. 4,238,213 are preferred for making the inventive fibers, other conventional methods may be employed such as sol-gel processes and extrusion through holes in precious metal alloy baskets.

The fibers so formed will have lengths in the range of from about 0.5 to 20 cm and diameters in the range of from about 0.05 to 10 microns with the average fiber diameter being in the range of about 1.5 to 3.5 microns. Table 2 shows the average fiber diameter (measured microscopically) and the unfiberized shot content of various inventive fibers. As may be seen, the average microscopic fiber diameter was 2.3 microns and the average unfiberized shot content was 27%.

For purposes of comparison, conventional mineral wool fibers were also tested with the results being given in Table 2 as numbers 226 and 229. These conventional fibers averaged 4.7 microns (measured microscopically) in diameter and had an average 40 wt% shot content. The continuous service temperature ranged from 1370°F to 1490°F, averaging 1420°F.

Table 3 contains an extensive chemical analysis of a number of inventive fibers. Because of the large number of fiber samples containing alumina additives made to the base calcium oxide/magnesia/silica system, only the average analysis of the minor constituent of these fibers are given in Table 3. The silica, alumina, magnesia, and calcium oxide contents for these fibers are given in Table 4.

As used herein, the "service temperature" of an inorganic fiber is determined by two parameters. The first is the obvious condition that the fiber must not soften or sinter at the temperature specified. It is this criterion which precludes the use of glass fibers at temperatures about 800°F to 1000°F (425° to 540°C). Additionally, a felt or blanket made from the fibers must not have excessive shrinkage when soaking at its service temperature. "Excess shrinkage" is usually defined to be a maximum of 5% linear or bulk shrinkage after prolonged exposure (usually for 24 hours) at the service temperature. Shrinkage of mats or blankets used as furnace liners and the like is of course a critical feature, for when the mats or blankets shrink they open fissures between them through which the heat can flow, thus defeating the purpose of the insulation. Thus, a fiber rated as a "1500°F (815°C) fiber" would be defined

as one which does not soften or sinter and which has acceptable shrinkage at that temperature, but which begins to suffer in one or more of the standard parameters at temperatures above 1500°F (815°C).

The service temperatures for a representative number of fibers in the inventive compositional range are listed in Table 2. The continuous service temperature for constant silica/magnesia/calcium oxide ratios are given in Table 6. As may be seen in all cases, the lower the alumina content of the fiber, the higher the service temperature will be, with the highest service temperature being at zero percent alumina for alumina contents less than 30%. Thus to attain the most desired properties of the inventive fiber it is not possible to accept any of the alumina contents resulting from melting the traditional mineral wool raw materials. Rather, various amounts of sufficiently pure oxides will be required to dilute the alumina contents to the desired low levels. To attain fibers of the highest service temperatures, only pure raw materials with essentially no significant amounts of alumina must be used.

A series of inventive fibers were also tested for their silicon extraction in a saline solution according to the following procedure:

A buffered model physiological saline solution was prepared by adding to 6 liters of distilled water the following ingredients at the indicated concentrations:

| Ingredient | Concentration, g/l |
|---|---|
| $MgCl_2 6H_2O$ | 0.160 |
| NaCl | 6.171 |

| | |
|---|---|
| KCl | 0.311 |
| $Na_2HPO_4$ | 0.149 |
| $Na_2SO_4$ | 0.079 |
| $CaCl_22H_2O$ | 0.060 |
| $NaHCO_3$ | 1.942 |
| $NaC_2H_3O_2$ | 1.066 |

Before testing, this solution was buffered to a pH of 7.6 by bubbling with a gaseous mixture of 5% $CO_2$/95%$N_2$.

One half (1/2) gram of each sample of fiber listed in Table 3 was then placed into separate closed, plastic bottles along with 50 cc of the prepared physiological saline solution and put into an ultrasonic bath for 5 hours. The ultrasonic vibration application was adjusted to give a temperature of 104°F at the end of the 5 hour period. At the end of the test period, the saline solution was filtered and the solution chemically analyzed for silicon content. The silicon concentration in the saline solution was taken to be a measure of the amount of fiber which solubilized during the 5 hour test period. The CaO and MgO contents of the fiber were similarly solubilized.

One of the inventive fibers was tested for silicon extraction in a physiological saline solution for periods of up to 6 months. Results were as follows:

| Fiber Number | Silicon Extraction in 6 Months | Steady State Silicon Extraction Rate For 0.20 $m^2/g$ Surface Area, % Si/day | Total Amphoteric Oxides in Fiber | Comments On Fiber Residue After 6 Months |
|---|---|---|---|---|
| 29 (inventive) | 96% | 0.16% | 1.0% | carbonate hydroxyl apatite fiber, disintegrated into small particles |
| 137 (non-inventive) | 3% | 0.013% | 8.9% | slight fine grained fibers with uniform corrosion |
| 235 (non-inventive) | 4% | 0.012% | 25.6% | no fiber corrosion; some surface deposition |

Categorization of oxides melts according to scales of acidity or basicity has been well known for many years. (See "A Scale of Acidity and Basicity in Glass," Glass Industry, February 1948, pp 73-74.) We have now found that by strictly controlling the compositions of the oxide melts according to the acidic or basicity behavior of the respective oxides, fibers can be made which are surprisingly soluble in saline solutions. Increasing the content of silica, alumina, and the amphoteric oxides in the fiber increases the acid ratio of the fiber composition. This tends to stabilize the system against silicon extraction by weak solutions as a result of relative changes in the interatomic bonding forces and extension of the silica network. Other amphoteric oxides besides alumina will have an alumina equivalency with respect to extraction by saline solutions. The amphoteric oxides zirconia and titania appear to have an alumina equivalency of close to 1 to 1. We have found that in general for desired high saline solubility the amount of total amphoteric oxides must be kept below about 10% depending upon the amount of silica present. On the other hand, with the exception of iron and manganese oxides, the basic oxides can vary widely since their alumina equivalency is small. However, while iron and manganese oxides are generally considered to be basic in nature, their behavior with respect to saline solubility more closely relate to the amphoteric oxides, thus the amounts of iron and manganese oxides must be similarly limited.

Many of the fibers were tested for their fire resistance according to the following simulated fire rating test procedure:

For screening test purposes, a small furnace was constructed using an electrically heated flat-plate element at the back of the heat source. A 6 inch x 6 inch x 2 inch thick sample of 1 3/4 to 6 1/2 pcf density of each formulated fiber was mounted parallel with the element and 1 inch from it. Thermocouples were then positioned at the center of the fiber sample surfaces. A computer was used to control power via a simple on-off relay system to the heating element. The position of the relay was based on the reading of the thermocouple on the sample surface nearest the element and the programmed fire test heat-up schedule.

The furnace was heated so as to follow a standard ASTM E-119 time/temperature curve for the 2-hour test period. In the test utilized herein, failure of the fiber is considered to occur when the furnace is unable to maintain the standard temperature per ASTM E-119 because the fiber insulation has sintered sufficiently to allow heat to escape through the fiber layer.

The results of the testing of the fibers for saline solubility and the two hour ASTM E-119 fire test are given in Table 4 for the fibers made with alumina addition and in Table 5 for the remaining fibers to which other oxidic constituents were added. These additions included: $B_2O_3$, $P_2O_5$, $TiO_2$, $ZrO_2$, $Fe_2O_3$ + MnO, $La_2O_3$, $Cr_2O_3$, and $Na_2O$. For glass fibers within the scope of the invention to function in an ASTM E-119 fire test, i.e. to withstand the rising temperatures of a simulated fire which can reach 1850°F in two hours, it is necessary that they convert from an amorphous condition to a beneficial pseudo crystalline state during heat-up. The inventive fibers do this but can be assisted in this function by the inclusion of suitable crystal nucleating

agents. Such agents may include $TiO_2$, $ZrO_2$, platinum, $Cr_2O_3$, $P_2O_5$, and others. Such additions are within the scope of this invention.

TABLE 1
RAW MATERIALS USED

| | Pure Raw Materials | | | | |
|---|---|---|---|---|---|
| | Silica Sand | Quick Lime | Calcined Dolomite | Aluminum Oxide | Magnesium Oxide |
| ACIDIC OXIDES | | | | | |
| SiO2 | 99.0 | 0.34 | 0.50 | 0.02 | 0.4 |
| AMPHOTERIC OXIDES | | | | | |
| $TiO_2$ | nil | nil | nil | 0.002 | nil |
| $Al_2O_3$ | 0.30 | 0.26 | 0.50 | 98.8 | 0.1 |
| BASIC OXIDES | | | | | |
| $Fe_2O_3$ | 0.30 | 0.05 | 0.15 | 0.02 | 0.7 |
| MnO | -- | -- | -- | -- | -- |
| MgO | 0.02 | 0.14 | 40.0 | nil | 96.3 |
| CaO | 0.03 | 97.75 | 57.0 | 0.01 | 2.0 |
| $Na_2O$ | 0.04 | 0.02 | 0.01 | 0.30 | 0.02 |
| $K_2O$ | 0.01 | 0.01 | nil | 0.01 | 0.01 |
| MISCELLANEOUS | | | | | |
| $SO_3$ | -- | -- | 0.4 | -- | -- |
| $S^=$ | -- | -- | -- | -- | -- |
| C | -- | -- | -- | -- | -- |
| LOI | 0.2 | 0.7 | 3.0 | 0.20 | 1.8 |
| TOTAL | 99.90 | 99.27 | 101.56 | 99.36 | 101.33 |

TABLE 1
RAW MATERIALS USED (continued)

| | Less Pure Raw Materials | | | |
|---|---|---|---|---|
| | Kaolin | Blast Furnace Slag | Nepheline Syenite | Talc |
| ACIDIC OXIDES | | | | |
| $SiO_2$ | 50.5 | 35.16 | 61.3 | 61.2 |
| AMPHOTERIC OXIDES | | | | |
| $TiO_2$ | 1.61 | 0.62 | 0.003 | nil |
| $Al_2O_3$ | 43.6 | 12.88 | 23.4 | 0.7 |
| BASIC OXIDES | | | | |
| $Fe_2O_3$ | 0.80 | 0.20 | 0.07 | 0.85 |
| MnO | -- | 0.62 | -- | -- |
| MgO | 0.01 | 16.06 | 0.05 | 31.7 |
| CaO | 0.04 | 32.94 | 0.58 | 0.19 |
| $Na_2O$ | 0.06 | 0.45 | 9.60 | -- |
| $K_2O$ | 0.02 | 0.25 | 4.50 | -- |
| MISCELLANEOUS | | | | |
| $SO_3$ | -- | 0.28 | -- | -- |
| $S^=$ | -- | 1.03 | -- | -- |
| C | -- | 0.30 | -- | -- |
| LOI | 2.90 | -- | 0.62 | 5.0 |
| TOTAL | 99.54 | 100.79 | 100.12 | 99.0 |

Silica Sand: Ottawa Silica - Sil-co-Sil Grade 295

Quick Lime: Mississippi Lime - Pulverized Quick Lime

Calcined Dolomite: Ohio Lime NO. 16 Burnt Dolomitic Lime

Aluminum Oxide: Reynolds Calcined Alumina, RC-23

Magnesium Oxide: Baymag 56 Feed Grade

Kaolin: American Cyanamide Andersonville Kaolin

Blast Furnace Slag: Calumite Morrisville Slag

Nepheline Syenite: Indusmin Grad A400

Talc: Pfizer Grade MP4426

Additives:

Soda Ash: 58.3% $Na_2O$

Boric Acid: 55.5% $B_2O_3$

Magnetite Iron Concentrates: 98.5% Iron Oxides

Zircon: 66.2% $ZrO_2$

Manganese Oxide: 99% $MnO_2$

Titanium Dioxide: 99% $TiO_2$

Chromium Oxide: 99.5% $Cr_2O_3$

Lanthanum Carbonate: Moly Corp.

TABLE 3
COMPOSITION OF FIBERS

| TEST NO. | ACIDIC OXIDES $B_2O_3$ | $SiO_2$ | $P_2O_5$ | SUB TOTAL | AMPHOTERIC OXIDES $TiO_2$ | $Al_2O_3$ | $ZrO_2$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $Al_2O_3$ additions (minor constituents only) | | | | | | | | |
| 1 to | 0.00 | -- | 0.00 | -- | 0.01 | -- | 0.01 | 0.02 |
| | -- | -- | -- | -- | -- | -- | -- | -- |
| Composition of Fibers with $B_2O_3$ additions | | | | | | | | |
| 164 | 0.32 | 64.8 | -- | 65.12 | -- | 0.06 | -- | 0.06 |
| 165 | 0.52 | 63.9 | -- | 64.42 | -- | 1.20 | -- | 1.20 |
| 166 | 0.64 | 64.6 | -- | 65.24 | -- | 0.06 | -- | 0.06 |
| 167 | 0.82 | 64.5 | -- | 65.32 | -- | 0.06 | -- | 0.06 |
| 168 | 1.33 | 64.1 | -- | 65.43 | -- | 0.06 | -- | 0.06 |
| 169 | 1.37 | 64.1 | -- | 65.47 | -- | 0.06 | -- | 0.06 |
| 170 | 2.22 | 63.6 | -- | 65.82 | -- | 0.06 | -- | 0.06 |
| 171 | 8.41 | 59.6 | -- | 68.01 | -- | 0.06 | -- | 0.06 |
| Composition of Fibers with $P_2O_5$ additions | | | | | | | | |
| 2 | -- | 49.6 | 6.05 | 55.65 | 0.06 | 0.38 | 0.04 | 0.48 |
| Composition of Fibers with $TiO_2$ additions | | | | | | | | |
| 173 | -- | 48.6 | -- | 48.6 | 10.0 | 41.4 | -- | 51.4 |
| Composition of Fibers with $ZrO_2$ additions | | | | | | | | |
| 174 | -- | 63.5 | -- | 63.5 | .01 | 0.88 | 0.21 | 1.10 |
| 175 | -- | 59.2 | -- | 59.2 | -- | 0.33 | 0.40 | 0.73 |
| 176 | -- | 59.5 | -- | 59.5 | -- | 0.31 | 0.42 | 0.73 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | BASIC OXIDES $FeO_3$ | $MnO$ | $La_2O_3$ | $Cr_2O_3$ | $MgO$ | $Li_2O$ | $CaO$ | $BaO$ | $Na_2O$ | $K_2O$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $Al_2O_3$ additions (minor constituents only) | | | | | | | | | | | |
| 1 to | 0.06 | 0.02 | 0.00 | 0.02 | -- | 0.00 | -- | 0.04 | 0.04 | 0.01 | .19 |
| | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | |
| Composition of Fibers with $B_2O_3$ additions | | | | | | | | | | | |
| 164 | -- | -- | -- | -- | 8.7 | -- | 26.6 | -- | -- | -- | 35.3 |
| 165 | -- | -- | -- | -- | 8.6 | -- | 26.2 | -- | -- | -- | 34.8 |
| 166 | -- | -- | -- | -- | 8.7 | -- | 26.5 | -- | -- | -- | 35.2 |
| 167 | -- | -- | -- | -- | 8.7 | -- | 26.5 | -- | -- | -- | 35.2 |
| 168 | -- | -- | -- | -- | 8.6 | -- | 26.3 | -- | -- | -- | 34.9 |
| 169 | -- | -- | -- | -- | 8.6 | -- | 26.3 | -- | -- | -- | 34.9 |
| 170 | -- | -- | -- | -- | 8.5 | -- | 26.1 | -- | -- | -- | 34.6 |
| 171 | -- | -- | -- | -- | 8.0 | -- | 24.0 | -- | -- | -- | 32.0 |
| Composition of Fibers with $P_2O_5$ additions | | | | | | | | | | | |
| 2 | 0.21 | 0.00 | -- | 0.68 | 11.15 | 0.00 | 31.45 | 0.00 | 0.05 | 0.04 | 43.58 |
| Composition of Fibers with $TiO_2$ additions | | | | | | | | | | | |
| 173 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| Composition of Fibers with $ZrO_2$ additions | | | | | | | | | | | |
| 174 | -- | -- | -- | -- | 0.33 | -- | 35.55 | -- | .03 | .01 | 35.92 |
| 175 | -- | -- | -- | -- | 0.41 | -- | 39.1 | -- | -- | -- | 39.51 |
| 176 | -- | -- | -- | -- | 0.42 | -- | 39.1 | -- | -- | -- | 39.52 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | MISCELLANEOUS $SO_3$ | Misc. | SUB TOTAL | TOTAL |
|---|---|---|---|---|
| Composition of Fibers with $Al_2O_3$ additions (minor constituents only) | | | | |
| 1 to | .05/ | .02 | .07/ | .14 |
| | .20 | -- | .22 | .44 |
| Composition of Fibers with $B_2O_3$ additions | | | | |
| 164 | -- | -- | -- | 100.48 |
| 165 | -- | -- | -- | 100.42 |
| 166 | -- | -- | -- | 100.5 |
| 167 | -- | -- | -- | 100.58 |
| 168 | -- | -- | -- | 100.39 |
| 169 | -- | -- | -- | 100.43 |
| 170 | -- | -- | -- | 100.48 |
| 171 | -- | -- | -- | 100.07 |
| Composition of Fibers with $P_2O_5$ additions | | | | |
| 2 | -- | 0.02 | 0.02 | 99.73 |
| Composition of Fibers with $TiO_2$ additions | | | | |
| 173 | -- | -- | -- | 100.0 |
| Composition of Fibers with $ZrO_2$ additions | | | | |
| 174 | -- | -- | -- | 100.52 |
| 175 | -- | -- | -- | 99.44 |
| 176 | -- | -- | -- | 99.75 |

TABLE 3

COMPOSITION OF FIBERS

| TEST NO. | ACIDIC OXIDES $B_2O_3$ | $SiO_2$ | $P_2O_5$ | SUB TOTAL | AMPHOTERIC OXIDES $TiO_2$ | $Al_2O_3$ | $ZrO_2$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $ZrO_2$ additions (Cont.) | | | | | | | | |
| 177 | -- | 59.7 | -- | 59.7 | -- | 0.34 | 0.50 | 0.84 |
| 8 | -- | 60.0 | -- | 60.0 | -- | 0.36 | 0.54 | 0.90 |
| 179 | -- | 59.2 | -- | 59.2 | -- | 0.35 | 0.58 | 0.93 |
| 180 | -- | 54.3 | -- | 54.3 | .01 | 1.29 | 0.58 | 1.88 |
| 181 | -- | 59.2 | -- | 59.2 | -- | 0.32 | 0.83 | 1.15 |
| 182 | -- | 46.85 | -- | 46.85 | .02 | 2.03 | 0.84 | 2.89 |
| 182(a) | -- | 59.4 | -- | 59.4 | -- | 0.38 | 2.31 | 2.69 |
| 183 | -- | 59.05 | -- | 59.05 | -- | 0.30 | 2.65 | 2.95 |
| 184 | -- | 57.96 | -- | 57.96 | -- | 0.42 | 3.11 | 3.53 |
| 185 | -- | 57.8 | -- | 57.80 | -- | 0.56 | 3.12 | 3.68 |
| 186 | -- | 59.05 | -- | 59.05 | -- | 0.38 | 3.27 | 3.65 |
| 187 | -- | 56.88 | -- | 56.88 | -- | 0.32 | 3.30 | 3.62 |
| 188 | -- | 57.7 | -- | 57.7 | -- | 0.20 | 3.30 | 3.50 |
| 189 | -- | 58.19 | -- | 58.19 | -- | 0.39 | 3.36 | 3.75 |
| 190 | -- | 57.86 | -- | 57.86 | -- | 0.36 | 3.37 | 3.73 |
| 191 | -- | 58.6 | -- | 58.6 | -- | 0.58 | 3.67 | 4.25 |
| 192 | -- | 58.4 | -- | 58.4 | -- | 0.65 | 3.69 | 4.34 |
| 193 | -- | 56.65 | -- | 56.65 | .02 | 3.35 | 4.50 | 7.87 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | BASIC OXIDES | | | | | | | | | | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $FeO_3$ | MnO | $La_2O_3$ | $Cr_2O_3$ | MgO | $Li_2O$ | CaO | BaO | $Na_2O$ | $K_2O$ | |
| Composition of Fibers with $ZrO_2$ additions (Cont.) | | | | | | | | | | | |
| 177 | -- | -- | -- | -- | 0.46 | -- | 38.7 | -- | -- | -- | 39.16 |
| 8 | -- | -- | -- | -- | 0.48 | -- | 38.3 | -- | -- | -- | 38.78 |
| 179 | -- | -- | -- | -- | 0.98 | -- | 37.0 | -- | -- | -- | 37.98 |
| 180 | .09 | .01 | -- | -- | 10.20 | -- | 32.75 | .01 | .04 | .02 | 43.12 |
| 181 | -- | -- | -- | -- | 1.13 | -- | 36.6 | -- | -- | -- | 37.73 |
| 182 | .08 | .01 | -- | -- | 20.6 | -- | 29.2 | .03 | .05 | .01 | 49.98 |
| 182(a) | -- | -- | -- | -- | 2.06 | -- | 34.9 | -- | -- | -- | 36.96 |
| 183 | .06 | .00 | -- | .05 | 3.08 | -- | 34.84 | .00 | .03 | .01 | 38.07 |
| 184 | -- | -- | -- | -- | 3.55 | -- | 35.17 | -- | -- | -- | 38.72 |
| 185 | -- | -- | -- | -- | 3.74 | -- | 34.4 | -- | -- | -- | 38.14 |
| 186 | -- | -- | -- | -- | 2.57 | -- | 36.94 | -- | -- | -- | 39.51 |
| 187 | -- | -- | -- | -- | 4.00 | -- | 36.45 | -- | -- | -- | 40.45 |
| 188 | -- | -- | -- | -- | 3.00 | -- | 36.0 | -- | -- | -- | 39.0 |
| 189 | -- | -- | -- | -- | 3.26 | -- | 35.39 | -- | -- | -- | 38.65 |
| 190 | -- | -- | -- | -- | 3.22 | -- | 35.66 | -- | -- | -- | 38.88 |
| 191 | -- | -- | -- | -- | 2.72 | -- | 33.5 | -- | -- | -- | 36.22 |
| 192 | -- | -- | -- | -- | 2.59 | -- | 33.2 | -- | -- | -- | 35.79 |
| 193 | .05 | .00 | -- | .00 | 3.35 | -- | 31.9 | .00 | .05 | .01 | 35.36 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | MISCELLANEOUS $SO_3$ | Misc. | SUB TOTAL | TOTAL |
|---|---|---|---|---|
| Composition of Fibers with $ZrO_2$ additions (Cont.) | | | | |
| 177 | -- | -- | -- | 99.70 |
| 8 | -- | -- | -- | 99.68 |
| 179 | -- | -- | -- | 98.11 |
| 180 | -- | .01 | .01 | 99.31 |
| 181 | -- | -- | -- | 98.08 |
| 182 | -- | .02 | .02 | 99.74 |
| 182(a) | -- | -- | -- | 99.05 |
| 183 | -- | .02 | .02 | 100.09 |
| 184 | -- | -- | -- | 100.21 |
| 185 | -- | -- | -- | 99.62 |
| 186 | -- | -- | -- | 102.21 |
| 187 | -- | -- | -- | 100.95 |
| 188 | -- | -- | -- | 100.20 |
| 189 | -- | -- | -- | 100.59 |
| 190 | -- | -- | -- | 100.47 |
| 191 | -- | -- | -- | 99.07 |
| 192 | -- | -- | -- | 98.53 |
| 193 | -- | .01 | .01 | 99.89 |

TABLE 3
COMPOSITION OF FIBERS

| TEST NO. | ACIDIC OXIDES $B_2O_3$ | $SiO_2$ | $P_2O_5$ | SUB TOTAL | AMPHOTERIC OXIDES $TiO_2$ | $Al_2O_3$ | $ZrO_2$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $FeO_3$ and MnO additions | | | | | | | | |
| 194 | -- | 64.9 | -- | 64.9 | -- | 0.06 | -- | 0.06 |
| 195 | -- | 49.8 | -- | 49.8 | .01 | 18.0 | .01 | 18.02 |
| 196 | -- | 50.4 | -- | 50.4 | .03 | 7.45 | .01 | 7.49 |
| 197 | -- | 64.34 | -- | 64.34 | -- | 0.06 | -- | 0.06 |
| 198 | -- | 63.70 | -- | 63.70 | -- | 1.20 | -- | 1.20 |
| 199 | -- | 63.54 | -- | 63.54 | -- | 1.20 | -- | 1.20 |
| 200 | -- | 38.9 | -- | 38.9 | .01 | 6.70 | .01 | 6.72 |
| 201 | -- | 64.3 | -- | 64.3 | -- | 0.06 | -- | 0.06 |
| 202 | -- | 44.6 | -- | 44.6 | .01 | 0.92 | .01 | 0.94 |
| 203 | -- | 63.3 | -- | 63.3 | -- | 1.15 | -- | 1.15 |
| 204 | -- | 63.6 | -- | 63.6 | -- | 0.06 | -- | 0.06 |
| 205 | -- | 43.8 | -- | 43.8 | .01 | 15.26 | .01 | 15.28 |
| 206 | -- | 62.3 | -- | 62.3 | -- | 1.20 | -- | 1.20 |
| 207 | -- | 63.3 | -- | 63.3 | -- | 0.06 | -- | 0.06 |
| 208 | -- | 43.9 | -- | 43.9 | .01 | 14.3 | .01 | 14.32 |
| 209 | -- | 62.0 | -- | 62.0 | -- | 0.06 | -- | 0.06 |
| 210 | -- | 60.0 | -- | 60.0 | -- | 2.0 | -- | 2.0 |
| 211 | -- | 60.0 | -- | 60.0 | -- | -- | -- | -- |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | BASIC OXIDES | | | | | | | | | | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $FeO_3$ | MnO | $La_2O_3$ | $Cr_2O_3$ | MgO | $Li_2O$ | CaO | BaO | $Na_2O$ | $K_2O$ | |
| Composition of Fibers with $FeO_3$ and MnO additions | | | | | | | | | | | |
| 194 | 0.06 | -- | -- | -- | 8.72 | -- | 26.6 | -- | -- | -- | 35.38 |
| 195 | .22 | -- | -- | -- | 0.2 | -- | 31.5 | -- | -- | -- | 31.92 |
| 196 | .48 | .04 | -- | -- | 15.2 | -- | 26.2 | -- | .07 | .05 | 42.04 |
| 197 | .50 | -- | -- | -- | 7.80 | -- | 26.4 | -- | -- | -- | 34.7 |
| 198 | .69 | -- | -- | -- | 7.73 | -- | 25.30 | -- | -- | -- | 33.72 |
| 199 | .72 | -- | -- | -- | 7.70 | -- | 25.04 | -- | -- | -- | 33.46 |
| 200 | .80 | -- | -- | -- | 16.1 | -- | 37.5 | -- | -- | -- | 54.40 |
| 201 | .96 | -- | -- | -- | 8.6 | -- | 26.4 | -- | -- | -- | 35.96 |
| 202 | 1.02 | -- | -- | -- | 18.1 | -- | 32.8 | -- | -- | -- | 51.92 |
| 203 | 1.61 | -- | -- | -- | 7.98 | -- | 25.4 | -- | -- | -- | 34.99 |
| 204 | 1.92 | -- | -- | -- | 8.6 | -- | 26.1 | -- | -- | -- | 36.62 |
| 205 | 2.90 | .04 | -- | .14 | 22.7 | -- | 15.05 | -- | .10 | .01 | 40.94 |
| 206 | 3.05 | -- | -- | -- | 8.0 | -- | 25.0 | -- | -- | -- | 36.05 |
| 207 | 3.45 | -- | -- | -- | 8.0 | -- | 25.5 | -- | -- | -- | 36.95 |
| 208 | 3.50 | -- | -- | -- | 24.4 | -- | 13.7 | -- | -- | -- | 41.6 |
| 209 | 4.81 | -- | -- | -- | 8.0 | -- | 25.5 | -- | -- | -- | 38.31 |
| 210 | -- | 8.0 | -- | -- | 30.0 | -- | -- | -- | -- | -- | 38.0 |
| 211 | -- | 20.0 | -- | -- | 20.0 | -- | -- | -- | -- | -- | 40.0 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | MISCELLANEOUS $SO_3$ | Misc. | SUB TOTAL | TOTAL |
|---|---|---|---|---|
| Composition of Fibers with $FeO_3$ and MnO additions | | | | |
| 194 | -- | -- | -- | 100.34 |
| 195 | .05 | .02 | .07 | 99.81 |
| 196 | .05 | .02 | .07 | 100.00 |
| 197 | -- | -- | -- | 99.1 |
| 198 | -- | -- | -- | 98.62 |
| 199 | -- | -- | -- | 98.20 |
| 200 | .05 | .02 | .07 | 100.09 |
| 201 | -- | -- | -- | 100.32 |
| 202 | -- | -- | -- | 97.46 |
| 203 | -- | -- | -- | 99.44 |
| 204 | -- | -- | -- | 100.28 |
| 205 | .05 | .08 | .13 | 100.15 |
| 206 | -- | -- | -- | 99.55 |
| 207 | -- | -- | -- | 100.31 |
| 208 | -- | -- | -- | 99.82 |
| 209 | -- | -- | -- | 100.37 |
| 210 | -- | -- | -- | 100.0 |
| 211 | -- | -- | -- | 100.0 |

TABLE 3
COMPOSITION OF FIBERS

| TEST NO. | ACIDIC OXIDES $B_2O_3$ | $SiO_2$ | $P_2O_5$ | SUB TOTAL | AMPHOTERIC OXIDES $TiO_2$ | $Al_2O_3$ | $ZrO_2$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $La_2O_3$ additions | | | | | | | | |
| -- | -- | 58.1 | -- | 58.1 | -- | 0.06 | -- | 0.06 |
| 213 | -- | 57.8 | -- | 57.8 | -- | 0.06 | -- | 0.06 |
| 214 | -- | 57.5 | -- | 57.5 | -- | 0.06 | -- | 0.06 |
| 215 | -- | 56.9 | -- | 56.9 | -- | 0.06 | -- | 0.06 |
| Composition of Fibers with $Cr_2O_3$ additions | | | | | | | | |
| 216 | -- | 62.6 | -- | 62.6 | 0.01 | 0.49 | 0.01 | 0.51 |
| Composition of Fibers with $Na_2O$ additions | | | | | | | | |
| 17 | -- | 64.7 | -- | 64.7 | -- | 0.06 | -- | 0.06 |
| 218 | -- | 64.5 | -- | 64.5 | -- | 0.06 | -- | 0.06 |
| 219 | -- | 64.4 | -- | 64.4 | -- | 0.06 | -- | 0.06 |
| 220 | -- | 63.5 | -- | 63.5 | -- | 1.20 | -- | 1.20 |
| 221 | -- | 64.3 | -- | 64.3 | -- | 0.06 | -- | 0.06 |
| 222 | -- | 64.2 | -- | 64.2 | -- | 0.06 | -- | 0.06 |
| 223 | -- | 64.0 | -- | 64.0 | -- | 0.06 | -- | 0.06 |
| 224 | -- | 63.0 | -- | 63.0 | -- | 0.06 | -- | 0.06 |
| 225 | -- | 60.3 | -- | 60.3 | -- | 0.06 | -- | 0.06 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | BASIC OXIDES $FeO_3$ | MnO | $La_2O_3$ | $Cr_2O_3$ | MgO | $Li_2O$ | CaO | BaO | $Na_2O$ | $K_2O$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Composition of Fibers with $La_2O_3$ additions | | | | | | | | | | | |
| -- | 0.16 | -- | 0.00 | -- | 4.60 | -- | 36.71 | -- | -- | -- | 41.47 |
| 213 | 0.15 | -- | 0.56 | -- | 4.58 | -- | 36.53 | -- | -- | -- | 41.82 |
| 214 | 0.15 | -- | 0.72 | -- | 4.55 | -- | 36.3 | -- | -- | -- | 41.72 |
| 215 | 0.15 | -- | 0.92 | -- | 4.51 | -- | 36.0 | -- | -- | -- | 41.58 |
| Composition of Fibers with $Cr_2O_3$ additions | | | | | | | | | | | |
| 216 | 0.08 | .00 | -- | 0.09 | 2.30 | -- | 34.10 | 0.00 | 0.03 | 0.01 | 36.61 |
| Composition of Fibers with $Na_2O$ additions | | | | | | | | | | | |
| 17 | -- | -- | -- | -- | 8.7 | -- | 26.6 | -- | 0.28 | -- | 35.58 |
| 218 | -- | -- | -- | -- | 8.7 | -- | 26.5 | -- | 0.45 | -- | 35.65 |
| 219 | -- | -- | -- | -- | 8.6 | -- | 26.5 | -- | 0.71 | -- | 35.80 |
| 220 | -- | -- | -- | -- | 8.5 | -- | 26.1 | -- | 0.87 | -- | 35.70 |
| 221 | -- | -- | -- | -- | 8.5 | -- | 26.2 | -- | 0.93 | -- | 35.63 |
| 222 | -- | -- | -- | -- | 8.6 | -- | 26.4 | -- | 1.11 | -- | 36.11 |
| 223 | -- | -- | -- | -- | 8.6 | -- | 26.3 | -- | 1.40 | -- | 36.3 |
| 224 | -- | -- | -- | -- | 8.5 | -- | 25.9 | -- | 2.60 | -- | 37.0 |
| 225 | -- | -- | -- | -- | 8.1 | -- | 24.8 | -- | 6.84 | -- | 39.74 |

TABLE 3
COMPOSITION OF FIBERS (continued)

| TEST NO. | MISCELLANEOUS SO$_3$ | Misc. | SUB TOTAL | TOTAL |
|---|---|---|---|---|
| Composition of Fibers with La$_2$ O$_3$ additions | | | | |
| -- | -- | -- | -- | 99.63 |
| 213 | -- | -- | -- | 99.68 |
| 214 | -- | -- | -- | 99.28 |
| 215 | -- | -- | -- | 98.54 |
| Composition of Fibers with Cr$_2$O$_3$ additions | | | | |
| 216 | -- | -- | -- | 99.72 |
| Composition of Fibers with Na$_2$O additions | | | | |
| 17 | -- | -- | -- | 100.34 |
| 218 | -- | -- | -- | 100.21 |
| 219 | -- | -- | -- | 100.26 |
| 220 | -- | -- | -- | 100.40 |
| 221 | -- | -- | -- | 99.99 |
| 222 | -- | -- | -- | 100.37 |
| 223 | -- | -- | -- | 100.36 |
| 224 | -- | -- | -- | 100.06 |
| 225 | -- | -- | -- | 100.1 |

TABLE 3

COMPOSITION OF FIBERS

| TEST NO. | ACIDIC OXIDES $B_2O_3$ | $SiO_2$ | $P_2O_5$ | SUB TOTAL | AMPHOTERIC OXIDES $TiO_2$ | $Al_2O_3$ | $ZrO_2$ | SUB TOTAL |
|---|---|---|---|---|---|---|---|---|
| Composition of Conventional Mineral Wools | | | | | | | | |
| 226 | - | 40.0 | - | 40.0 | 0.37 | 9.1 | 0.03 | 9.50 |
| | - | 39.9 | 0.02 | 39.92 | 1.11 | 12.85 | 0.03 | 13.99 |
| 228 | - | 37.65 | 0.84 | 38.49 | 2.35 | 9.85 | 0.04 | 12.24 |
| 229 | - | 41.75 | 0.12 | 41.87 | 1.07 | 16.0 | 0.03 | 17.10 |
| Composition of Refractory Fibers (Fibers with less than 25% Basic Oxides) | | | | | | | | |
| 231 | - | 31.0 | - | 31.0 | - | 47.5 | 0.02 | 47.52 |
| 232 | - | 37.1 | - | 37.1 | - | 59.2 | - | 59.2 |
| 233 | - | 50.0 | - | 50.0 | - | 40.0 | - | 40.0 |
| 234 | - | 54.0 | - | 54.0 | - | 46.0 | - | 46.0 |
| 235 | - | 58.47 | 1.15 | 59.62 | 0.98 | 24.54 | 0.03 | 25.55 |
| 236 | - | 52.1 | - | 52.1 | 1.76 | 44.4 | .23 | 46.39 |
| 237 | - | 52.0 | - | 52.0 | 1.71 | 42.2 | 2.93 | 46.84 |
| 238 | - | 49.8 | - | 49.8 | 1.60 | 38.3 | 9.32 | 49.22 |
| 239 | - | 48.6 | - | 48.6 | 1.55 | 36.2 | 12.3 | 50.05 |
| 240 | - | 47.8 | - | 47.8 | 1.50 | 34.4 | 15.1 | 51.00 |
| 241 | - | 46.2 | - | 46.2 | 1.40 | 31.0 | 20.7 | 53.10 |
| 242 | - | 28 | - | 28 | 19 | 50 | 3 | 72 |
| 243 | - | 64.5 | - | 64.5 | - | 27.4 | - | 27.4 |

SUBSTITUTE SHEET

TABLE 3 (cont'd.)

COMPOSITION OF FIBERS

| | BASIC OXIDES | | | | | | | | | | | | MISCELLANEOUS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TEST NO. | $FeO_3$ | MnO | $La_2O_3$ | $Cr_2O_3$ | MgO | $Li_2O$ | CaO | BaO | $Na_2O$ | $K_2O$ | SUB TOTAL | $SO_3$ | Misc. | SUB TOTAL | TOTAL |
| Composition of Conventional Mineral Wools | | | | | | | | | | | | | | | |
| 226 | 0.47 | 0.64 | - | 0.02 | 11.2 | 0.01 | 36.5 | 0.04 | 0.54 | 0.55 | 49.97 | 0.1 | 0.59 | 0.69 | 100.16 |
| | 0.35 | 0.24 | - | 0.00 | 6.05 | 0.01 | 38.55 | 0.12 | 0.23 | 0.27 | 45.82 | 0.67 | 0.07 | 0.74 | 100.47 |
| 228 | 9.7 | 0.22 | - | 0.04 | 12.95 | 0.01 | 23.55 | 0.07 | 2.01 | 0.80 | 49.35 | 0.42 | 0.19 | 0.61 | 100.69 |
| 229 | 3.75 | 0.23 | - | 0.02 | 6.45 | 0.63 | 27.75 | 0.03 | 2.04 | 0.63 | 41.53 | 0.56 | 0.08 | 0.64 | 101.14 |
| Composition of Refractory Fibers (Fibers with less than 25% Basic Oxides | | | | | | | | | | | | | | | |
| 231 | - | - | - | - | - | - | 1.2 | - | 20.2 | - | 21.4 | - | - | - | 99.92 |
| 232 | - | - | - | - | - | - | 0.2 | - | 3.1 | - | 3.3 | - | - | - | 99.6 |
| 233 | - | - | - | - | - | - | 5.6 | - | 4.4 | - | 10.0 | - | - | - | 100 |
| 234 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | 100 |
| 235 | 3.70 | 0.02 | - | 0.00 | 1.44 | 0.02 | 5.78 | 0.54 | 1.55 | 1.18 | 14.23 | 0.47 | 0.24 | 0.71 | 100.11 |
| 236 | .83 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | 1.13 | - | - | - | 99.62 |
| 237 | .77 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | 1.07 | - | - | - | 99.91 |
| 238 | .72 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | 1.02 | - | - | - | 100.04 |
| 239 | .70 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | 1.00 | - | - | - | 99.65 |
| 240 | .68 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | .98 | - | - | - | 99.78 |
| 241 | .63 | - | - | - | 0.07 | - | 0.12 | - | .05 | .06 | 0.93 | - | - | - | 100.23 |
| 242 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | 100 |
| 243 | - | - | - | - | - | 8.4 | - | - | - | - | 8.4 | - | - | - | 100.3 |

TABLE 4

TEST RESULTS ON FIBERS MADE WITH ALUMINA ADDITIONS

| NO. | COMPOSITION, WT% Acidic Oxides $SiO_2$ | Amphoteric Oxides $Al_2O_3$ | Amphoteric Oxides Total | Basic Oxides CaO | Basic Oxides MgO | Basic Oxides Total | Total Analytical | 5 Hour Saline Extraction ppm. Si | E-119 Fire Test Thickness Density | E-119 Fire Test 2 Hour Test** |
|---|---|---|---|---|---|---|---|---|---|---|
| 0 to 1 1/2% Amphoteric Oxides | | | | | | | | | | |
| 1 | 32 | 0.2 | 0.22 | 39 | 29 | 68.1 | 100.37 | * | * | * |
| 2 | 31.3 | 0.2 | 0.22 | 33.3 | 35.5 | 68.9 | 100.47 | * | * | * |
| 3 | 41.9 | 0.28 | 0.30 | 57.5 | 0.1 | 57.7 | 99.95 | 80 | - | - |
| 4 | 43.5 | 0.33 | 0.35 | 46.0 | 10.4 | 56.5 | 100.40 | 58 | - | - |
| 5 | 43.7 | 0.25 | 0.27 | 39.8 | 16.6 | 56.5 | 100.52 | 46 | 2.0/1.27 | F |
| 6 | 45.0 | 0.50 | 0.52 | 54.4 | 0.1 | 54.6 | 100.17 | 75 | - | - |
| 7 | 46.5 | 0.20 | 0.22 | 9.2 | 45.1 | 54.4 | 101.17 | * | * | * |
| 8 | 48.2 | 0.20 | 0.22 | 5.0 | 47.6 | 52.7 | 101.17 | * | * | * |
| 9 | 47.9 | 0.22 | 0.24 | 19.3 | 33.5 | 52.9 | 101.09 | 50 | - | - |
| 10 | 48.5 | 0.56 | 0.58 | 8.8 | 43.0 | 51.9 | 101.03 | 51 | - | - |
| 11 | 48.6 | 0.56 | 0.58 | 13.3 | 38.3 | 51.7 | 100.93 | 46 | - | - |
| 12 | 49.2 | 0.42 | 0.44 | 28.0 | 22.9 | 51.0 | 100.69 | 67 | - | - |
| 13 | 49.2 | 0.17 | 0.19 | 3.4 | 48.3 | 51.8 | 101.24 | * | * | * |
| 14 | 50.0 | 0.10 | 0.12 | 7.0 | 43.0 | 50.1 | 100.27 | 56 | - | - |
| 15 | 50.7 | 0.10 | 0.12 | 15.7 | 33.7 | 49.5 | 100.37 | 60 | - | - |
| 16 | 51.1 | 0.45 | 0.47 | 29.8 | 19.0 | 48.9 | 100.52 | 65 | - | - |
| 17 | 51.2 | 0.33 | 0.35 | 39.7 | 9.0 | 48.8 | 100.40 | 51 | 2.0/2.59 | F |
| 18 | 53.2 | 0.64 | 0.66 | 2.8 | 44.3 | 47.2 | 101.11 | 56 | - | F |
| 19 | 53.4 | 0.28 | 0.30 | 45.6 | 0.1 | 45.8 | 99.55 | 77 | 2.0/1.97 | F |

* = Not Fiberizable ** P = Pass, F = Failed

## EXPERIMENTAL DATA

| | COMPOSITION, WT% | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | Total | 5 Hour Saline Extraction | E-119 Fire Test | |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Analytical | ppm. Si | Thickness Density | 2 Hour Test** |
| 0 to 1 1/2% Amphoteric Oxides | | | | | | | | | | |
| 20 | 53.8 | 0.33 | 0.35 | 35.1 | 10.8 | 46.0 | 100.20 | 83 | 2.0/1.97 | F |
| 21 | 53.9 | 0.40 | 0.42 | 25.5 | 20.5 | 46.1 | 100.47 | 68 | _ | _ |
| 22 | 54.5 | 1.00 | 1.02 | 7.5 | 36.5 | 44.1 | 99.67 | 30 | _ | _ |
| 23 | 55.9 | 0.08 | 0.10 | 43.0 | 0.45 | 43.55 | 99.60 | 51 | 2.0/1.94 | F |
| 24 | 56.0 | 0.40 | 0.42 | 27.0 | 17.0 | 44.1 | 100.57 | 69 | 2.0/2.12 | F |
| 25 | 56.35 | 0.20 | 0.24 | 34.4 | 8.25 | 42.75 | 99.39 | 70 | 2.0/1.87 | F |
| 26 | 56.4 | 0.91 | 0.93 | 35.1 | 7.39 | 42.59 | 99.97 | 47 | _ | _ |
| 27 | 57.0 | 1.03 | 1.05 | 24.5 | 17.6 | 42.2 | 100.30 | 46 | _ | _ |
| 28 | 57.0 | 1.09 | 1.11 | 35.0 | 6.84 | 41.94 | 100.10 | 40 | _ | _ |
| 29 | 57.25 | 0.92 | 0.94 | 36.95 | 3.95 | 41.1 | 99.56 | 56 | 1.88/2.20 | F |
| 30 | 57.8 | 0.75 | 0.78 | 34.75 | 6.2 | 41.05 | 99.85 | _ | 2.0 /1.97 | F |
| 31 | 58.1 | 0.03 | 0.05 | 36.7 | 4.53 | 41.33 | 99.53 | 59 | 2.0 /1.91 | F |
| 32 | 58.2 | 1.08 | 1.10 | 35.7 | 4.79 | 40.59 | 99.94 | 80 | 2.0 /1.91 | F |
| 33 | 58.3 | 0.03 | 0.05 | 40.8 | 0.31 | 41.21 | 99.61 | 49 | 2.0 /1.91 | F |
| 34 | 58.4 | 0.37 | 0.39 | 15.3 | 26.3 | 41.7 | 100.54 | 61 | 2.0 /1.91 | F |
| 35 | 58.6 | 0.09 | 0.11 | 35.0 | 5.36 | 40.46 | 99.22 | 74 | 2.0 /1.94 | P |
| 36 | 58.7 | 0.05 | 0.07 | 40.2 | 0.27 | 40.57 | 99.39 | 58 | 2.0 /1.91 | F |
| 37 | 58.5 | 0.49 | 0.53 | 34.4 | 5.6 | 40.1 | 99.32 | 59 | 2.0 /2.01 | P |
| 38 | 58.8 | 0.41 | 0.43 | 35.4 | 6.2 | 41.7 | 100.98 | 56 | _ | _ |

* = Not Fiberizable ** P = Poor, F = Failed

**EXPERIMENTAL DATA**

| | COMPOSITION, WT% | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | | 5 Hour Saline Extraction | E-119 Fire Test | |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Total Analytical | ppm. Si | Thickness Density | 2 Hour Test** |
| 0 to 1 1/2% Amphoteric Oxides | | | | | | | | | | |
| 39 | 58.9 | 0.08 | 0.10 | 34.2 | 6.10 | 40.4 | 99.45 | 67 | 2.0/1.86 | P |
| 40 | 59.0 | 0.24 | 0.26 | 35.9 | 3.8 | 39.9 | 99.21 | 49 | 2.0/1.97 | P |
| 41 | 59.1 | 0.09 | 0.11 | 40.3 | 0.43 | 40.83 | 100.09 | 68 | 2.0/1.90 | P |
| 42 | 59.2 | 0.24 | 0.26 | 4.7 | 36.8 | 41.60 | 101.11 | 47 | 2.5/1.4 | F |
| 43 | 59.15 | 0.32 | 0.34 | 35.55 | 4.75 | 40.40 | 99.94 | 60 | 2.0/1.95 | P |
| 44 | 59.4 | 0.04 | 0.06 | 29.8 | 10.7 | 40.60 | 100.11 | 61 | 2.0/1.92 | P |
| 45 | 59.5 | 0.02 | 0.04 | 34.2 | 5.98 | 40.28 | 99.87 | 77 | 2.0/1.90 | P |
| 46 | 59.5 | 0.02 | 0.04 | 32.1 | 8.16 | 40.36 | 99.95 | 73 | 2.0/1.89 | F |
| 47 | 59.6 | 1.43 | 1.45 | 22.5 | 16.8 | 39.6 | 100.8 | 51 | 2.0/1.88 | F |
| 48 | 59.6 | 0.03 | 0.05 | 28.7 | 11.4 | 40.2 | 99.9 | 70 | 2.0/1.91 | P |
| 50 | 59.8 | 0.28 | 0.30 | 40.5 | 0.11 | 40.71 | 100.86 | 30 | 2.0/2.01 | P |
| 51 | 59.9 | 1.48 | 1.50 | 25.8 | 12.9 | 39.0 | 100.55 | 47 | 2.0/1.98 | P |
| 52 | 59.9 | 1.31 | 1.33 | 28.1 | 11.0 | 39.4 | 100.78 | 45 | 2.0/1.95 | P |
| 53 | 60.0 | 1.41 | 1.43 | 22.3 | 16.4 | 39.0 | 100.58 | 41 | 2.0/1.91 | P |
| 54 | 60.3 | 0.17 | 0.19 | 32.3 | 6.36 | 38.76 | 99.30 | 59 | 2.0/1.89 | P |
| 55 | 60.4 | 1.05 | 1.07 | 28.5 | 9.85 | 38.45 | 99.97 | 45 | 2.0/1.95 | P |
| 56 | 60.5 | 1.11 | 1.13 | 27.9 | 10.7 | 38.9 | 100.68 | 36 | 2.0/1.94 | F |
| 57 | 60.7 | 0.93 | 0.95 | 28.7 | 9.47 | 38.27 | 99.97 | 51 | 2.0/1.93 | P |
| 58 | 60.8 | 0.2 | 0.22 | 36. | 3. | 39.10 | 100.17 | 56 | – | – |

* = Not Fiberizable ** P = Poor, F = Failed

**EXPERIMENTAL DATA**

| | COMPOSITION, WT% | | | | | | | 5 Hour | E-119 Fire Test | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | Total | Saline Extraction | Thickness | 2 Hour |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Analytical | ppm. Si | Density | Test** |
| 0 to 1 1/2% Amphoteric Oxides | | | | | | | | | | |
| 59 | 61.7 | 0.02 | 0.04 | 32.6 | 5.19 | 37.89 | 99.68 | 65 | 2.0/1.97 | P |
| 60 | 62.4 | 0.04 | 0.06 | 21.7 | 15.5 | 37.3 | 99.81 | 76 | 2.0/1.88 | P |
| 61 | 62.5 | 0.02 | 0.04 | 30.3 | 6.64 | 37.04 | 99.63 | 66 | 2.0/1.92 | P |
| 62 | 62.5 | 0.03 | 0.05 | 29.5 | 7.70 | 37.30 | 99.90 | 64 | 2.0/1.82 | P |
| 63 | 63.1 | 0.02 | 0.04 | 31.1 | 5.28 | 36.48 | 99.67 | 46 | 2.0/1.95 | P |
| 64 | 63.1 | 1.25 | 1.27 | 25.2 | 10.2 | 35.5 | 99.92 | 19 | 2.0/1.96 | P |
| 65 | 63.5 | 1.49 | 1.51 | 24.0 | 10.9 | 35.0 | 100.06 | 12 | 2.0/1.91 | P |
| 66 | 63.8 | 1.13 | 1.15 | 28.4 | 5.79 | 34.29 | 99.29 | 52 | 2.0/2.01 | P |
| 67 | 63.8 | 1.41 | 1.43 | 22.8 | 11.8 | 34.7 | 99.98 | 17 | 2.0/1.88 | P |
| 68 | 64.1 | 1.23 | 1.25 | 30.97 | 2.60 | 33.67 | 99.07 | 7 | 2.0/1.88 | P |
| 69 | 64.1 | 1.47 | 1.49 | 28.6 | 4.83 | 33.53 | 99.17 | 49 | 2.0/1.99 | P |
| 70 | 65.3 | 0.03 | 0.05 | 27.4 | 6.68 | 34.18 | 99.58 | 37 | 2.0/1.91 | P |
| 71 | 65.4 | 1.15 | 1.17 | 3.12 | 30.1 | 33.32 | 99.94 | 46 | 2.0/1.88 | F |
| 72 | 65.6 | 0.01 | 0.03 | 27.4 | 6.50 | 34.0 | 99.68 | 35 | 2.0/2.00 | P |
| 73 | 65.8 | 0.02 | 0.04 | 28.6 | 5.21 | 33.91 | 99.80 | 44 | — | — |
| 74 | 65.9 | 0.03 | 0.05 | 21.9 | 11.8 | 33.8 | 99.80 | 30 | 2.0/1.87 | P |
| 75 | 65.9 | 0.03 | 0.05 | 25.8 | 7.88 | 33.78 | 99.78 | 25 | 2.0/1.91 | P |
| 76 | 65.4 | 1.15 | 1.17 | 3.12 | 30.1 | 33.23 | 99.84 | 46 | 2.0/1.88 | F |

* = Not Fiberizable ** P = Poor, F = Failed

SUBSTITUTE SHEET

EXPERIMENTAL DATA

| | COMPOSITION, WT% | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | | 5 Hour Saline Extraction | E-119 Fire Test | |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Total Analytical | ppm. Si | Thickness Density | 2 Hour Test** |
| 0 to 1 1/2% Amphoteric Oxides (Cont.) | | | | | | | | | | |
| 77 | 66.1 | 0.59 | 0.61 | 4.02 | 28.7 | 33.02 | 99.88 | 50 | | F |
| 78 | 67.1 | - | - | 6.43 | 26.5 | 33.03 | 100.18 | 78 | - | F |
| 79 | 67.2 | 0.02 | 0.04 | 8.67 | 24.0 | 32.77 | 100.06 | 84 | 2.0/1.89 | F |
| 80 | 68.4 | - | - | 1.6 | 30.1 | 31.8 | 100.25 | * | 2.0/2.03 | F |
| 81 | 68.6 | 0.25 | 0.27 | 29.0 | 1.09 | 30.19 | 99.11 | 18 | * | * |
| 82 | 68.8 | - | - | 10.2 | 21.3 | 31.6 | 100.45 | 31 | 2.0/2.00 | P |
| 83 | 68.9 | 0.03 | 0.05 | 18.1 | 12.7 | 30.9 | 99.9 | 30 | - | - |
| 84 | 69.0 | | | 7.2 | 23.8 | 31.0 | 100.05 | 18 | 2.0/2.00 | P |
| | | | | | | | | | - | - |
| 1 1/2% to 3% Amphoteric Oxides | | | | | | | | | | |
| 85 | 50.0 | 2.00 | 2.02 | 5.0 | 43.0 | 48.1 | 100.17 | - | * | * |
| 86 | 52.6 | 2.00 | 2.02 | 3.8 | 41.7 | 45.6 | 100.27 | 51 | 2.0/1.88 | F |
| 87 | 56.1 | 2.41 | 2.43 | 30.3 | 10.6 | 41.0 | 99.58 | 39 | 2.0/1.89 | F |
| 88 | 56.2 | 1.82 | 1.84 | 24.4 | 17.3 | 41.8 | 99.89 | 65 | - | - |
| 89 | 58.1 | 2.01 | 2.03 | 3.83 | 36.3 | 40.43 | 100.71 | 44 | 2.0/1.99 | P |
| 90 | 58.9 | 2.26 | 2.28 | 36.6 | 1.4 | 38.1 | 99.33 | 18 | 2.0/1.82 | P |
| 91 | 59.0 | 2.93 | 2.95 | 36.3 | 1.0 | 37.4 | 99.40 | 9 | 2.0/1.87 | P |
| 92 | 59.4 | 0.38 | 2.69 | 34.9 | 2.1 | 37.1 | 99.24 | 25 | 2.0/2.06 | P |
| 93 | 59.8 | 2.54 | 2.56 | 27.4 | 10.0 | 37.5 | 99.91 | 11 | - | - |
| 94 | 60.1 | 1.68 | 1.70 | 28.0 | 9.9 | 38.0 | 99.85 | 29 | 2.0/1.98 | P |

* = Not Fiberizable

* P = Pass, F = Failed

## EXPERIMENTAL DATA

| NO. | COMPOSITION, WT% — Acidic Oxides $SiO_2$ | Amphoteric Oxides $Al_2O_3$ | Amphoteric Oxides Total | Basic Oxides CaO | Basic Oxides MgO | Basic Oxides Total | Total Analytical | 5 Hour Saline Extraction ppm. Si | E-119 Fire Test Thickness Density | E-119 Fire Test 2 Hour Test** |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 1/2% to 3% Amphoteric Oxides (Cont.) | | | | | | | | | | |
| 95 | 60.2 | 2.21 | 2.23 | 32.7 | 4.9 | 37.7 | 100.18 | 50 | 2.0/2.04 | P |
| 96 | 61.4 | 2.17 | 2.19 | 26.2 | 10.1 | 36.4 | 100.04 | 18 | 2.0/1.87 | P |
| 97 | 61.4 | 1.66 | 1.68 | 29.9 | 6.9 | 36.9 | 100.03 | 61 | 2.0/1.91 | P |
| 98 | 61.8 | 2.84 | 2.86 | 34.0 | 0.2 | 34.3 | 99.01 | 51 | 2.0/1.93 | P |
| 99 | 62.0 | 2.81 | 2.83 | 34.1 | 0.2 | 34.4 | 99.28 | 55 | 2.0/1.90 | P |
| 100 | 62.1 | 2.75 | 2.77 | 33.8 | 0.2 | 34.1 | 99.02 | 13 | 2.0/1.91 | P |
| 101 | 62.7 | 1.79 | 1.81 | 25.6 | 9.4 | 35.1 | 99.66 | 18 | 2.0/1.96 | P |
| 102 | 63.0 | 2.54 | 2.56 | 33.1 | 0.2 | 33.4 | 99.05 | 37 | 2.0/1.87 | P |
| 103 | 63.9 | 1.84 | 1.86 | 30.7 | 2.5 | 33.3 | 99.11 | 38 | 2.0/1.94 | P |
| 104 | 64.1 | 1.83 | 1.85 | 17.7 | 16.3 | 34.3 | 100.4 | 12 | 2.0/1.95 | P |
| 105 | 65.1 | 2.15 | 2.17 | 9.74 | 23.1 | 33.15 | 100.57 | 17 | - | P |
| 106 | 65.6 | 1.56 | 1.58 | 2.7 | 29.7 | 32.5 | 99.73 | 33 | 2.0/1.91 | P |
| 107 | 66.7 | 1.80 | 1.82 | 30.7 | 0.1 | 30.9 | 99.47 | 2 | 2.0/1.90 | P |
| 3 to 4% Amphoteric Oxides | | | | | | | | | | |
| 108 | 49.8 | 3.5 | 3.52 | 4.98 | 40.9 | 46.18 | 99.65 | 33 | - | - |
| 109 | 50.3 | 3.58 | 3.60 | 45.0 | 0.64 | 45.74 | 99.69 | 19 | 2.0/1.96 | F |
| 110 | 55.1 | 3.77 | 3.79 | 7.89 | 33.7 | 41.89 | 100.93 | 33 | 2.0/2.06 | P |

* = Not Fiberizable ** P = Pass, F = Failed

EXPERIMENTAL DATA

| | COMPOSITION, WT% | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | Total | 5 Hour Saline Extraction | E-119 Fire Test | |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Analytical | ppm. Si | Thickness Density | 2 Hour Test** |
| 3% to 4% Amphoteric Oxides (Cont.) | | | | | | | | | | |
| 111 | 55.6 | 0.24 | 3.66 | 37.1 | 4.65 | 41.85 | 101.16 | - | 2.0/2.12 | F |
| 112 | 56.5 | 0.35 | 3.65 | 36.51 | 4.17 | 40.78 | 100.98 | - | 2.0/1.99 | F |
| 113 | 56.7 | 3.52 | 3.54 | 23.5 | 16.2 | 39.8 | 100.09 | 19 | 2.0/1.89 | F |
| 114 | 56.7 | 3.06 | 3.08 | 23.4 | 16.6 | 40.28 | 100.11 | 40 | 2.0/4.02 | F |
| 115 | 56.88 | 0.32 | 3.64 | 36.45 | 4.00 | 40.45 | 101.02 | 51 | - | - |
| 115a | 57.5 | 3.29 | 3.31 | 37.7 | 0.75 | 38.55 | 99.41 | 6 | 2.0/1.93 | F |
| 116 | 58.1 | 3.05 | 3.07 | 25.6 | 12.8 | 38.5 | 99.72 | 20 | 2.0/1.9 | F |
| 117 | 58.2 | 3.75 | 3.77 | 36.4 | 0.67 | 37.17 | 99.19 | 38 | 2.0/2.0 | F |
| 119 | 58.80 | 3.76 | 3.78 | 36.7 | 0.24 | 37.04 | 99.67 | 28 | 2.0/1.97 | F |
| 120 | 61.2 | 3.77 | 3.79 | 34.0 | 0.24 | 34.34 | 99.38 | 18 | 2.0/1.94 | P |
| 4 to 6% Amphoteric Oxides | | | | | | | | | | |
| 121 | 49.7 | 4.04 | 4.06 | 26.4 | 19.6 | 46.1 | 99.91 | 37 | - | - |
| 122 | 55.8 | 5.20 | 5.22 | 30.1 | 9.2 | 39.4 | 100.47 | 7 | 2.0/1.88 | F |
| 123 | 56.85 | 5.40 | 5.41 | 31.8 | 5.65 | 37.55 | 99.91 | 4 | 2.0/1.99 | F |
| 124 | 57.0 | 4.68 | 4.70 | 22.0 | 15.6 | 37.7 | 99.45 | 32 | 2.0/2.00 | F |

* = Not Fiberizable    ** P = Pass, F = Failed

EXPERIMENTAL DATA

| | COMPOSITION, WT% | | | | | | | 5 Hour | E-119 Fire Test | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | Total | Saline Extraction | Thickness | 2 Hour |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Analytical | ppm. Si | Density | Test** |
| 6 to 8% Amphoteric Oxides | | | | | | | | | | |
| 125 | 39.2 | 6.90 | 6.92 | 38.5 | 14.0 | 52.6 | 98.72 | 37 | - | - |
| 126 | 46.9 | 7.66 | 7.68 | 44.8 | 0.3 | 45.2 | 99.83 | 6 | 2.0/1.97 | F |
| 127 | 49.3 | 6.40 | 6.42 | 25.3 | 18.4 | 43.8 | 99.57 | 19 | 2.0/2.0 | F |
| 128 | 50.4 | 7.45 | 7.48 | 26.2 | 15.2 | 41.5 | 99.43 | 18 | 2.0/3.17 | F |
| 129 | 54.7 | 7.60 | 7.62 | 30.7 | 6.5 | 37.3 | 99.67 | 7 | 2.0/1.98 | F |
| 130 | 56.1 | 6.34 | 6.36 | 30.6 | 6.9 | 37.6 | 100.11 | 4 | 2.0/2.04 | F |
| 131 | 57.9 | 6.7 | 6.72 | 5.9 | 29.7 | 35.6 | 100.27 | 2 | - | - |
| 132 | 58.5 | 6.16 | 6.18 | 31.2 | 4.0 | 35.2 | 99.93 | 2 | 2.0/2.01 | F |
| 133 | 59.7 | 7.08 | 7.10 | 27.9 | 5.1 | 33.1 | 99.9 | 2 | 2.0/2.04 | F |
| 8 to 10% Amphoteric Oxides | | | | | | | | | | |
| 134 | 38.6 | 9.3 | 9.32 | 38.4 | 13.7 | 52.2 | 100.17 | 12 | - | - |
| 135 | 42.8 | 8.8 | 9.13 | 36.7 | 9.6 | 46.76 | 98.69 | 13 | - | - |
| 136 | 44.5 | 8.76 | 8.78 | 45.5 | 0.52 | 46.12 | 99.45 | 3 | - | - |
| 137 | 52.1 | 8.9 | 8.92 | 23.7 | 16.2 | 40.0 | 101.02 | 1.2 | - | - |
| 138 | 52.5 | 9.67 | 9.69 | 33.5 | 4.21 | 37.81 | 100.05 | 1.0 | 2.0/1.99 | F |
| 139 | 53.7 | 8.7 | 8.72 | 22.5 | 16.3 | 38.9 | 101.37 | 1.7 | - | - |
| 140 | 56.6 | 9.2 | 9.22 | 23.5 | 10.9 | 34.5 | 100.37 | 1.2 | 2.0/2.05 | F |

* = Not Fiberizable ** P = Pass, F = Failed

SUBSTITUTE SHEET

## EXPERIMENTAL DATA

| | COMPOSITION, WT% | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acidic Oxides | Amphoteric Oxides | | Basic Oxides | | | Total | 5 Hour Saline Extraction | E-119 Fire Test | |
| NO. | $SiO_2$ | $Al_2O_3$ | Total | CaO | MgO | Total | Analytical | ppm. Si | Thickness Density | 2 Hour Test** |
| 10 to 12% Amphoteric Oxides | | | | | | | | | | |
| 141 | 41.0 | 10.05 | 10.07 | 48.25 | 0.3 | 48.70 | 99.87 | 6 | 2.0/2.00 | F |
| 142 | 51.3 | 10.9 | 10.92 | 37.2 | 0.2 | 37.5 | 99.77 | 0.8 | 2.0/2.04 | F |
| 143 | 52.4 | 10.7 | 10.72 | 23.1 | 16.1 | 39.3 | 102.42 | 0.7 | 2.0/2.00 | F |
| 144 | 52.7 | 10.2 | 10.22 | 22.1 | 16.0 | 38.2 | 101.12 | 0.5 | - | - |
| 12 to 20% Amphoteric Oxides | | | | | | | | | | |
| 145 | 41.5 | 13.0 | 13.02 | 44.2 | 0.5 | 44.8 | 99.37 | 1.2 | - | - |
| 146 | 49.8 | 18.0 | 18.02 | 31.5 | 0.2 | 32.02 | 99.89 | 0.5 | - | - |
| 147 | 55.6 | 12.9 | 12.92 | 13.2 | 18.4 | 31.7 | 100.27 | 1.8 | 2.0/2.54 | F |
| 20 to 30% Amphoteric Oxides | | | | | | | | | | |
| 148 | 36.5 | 28.4 | 28.42 | 34.4 | 0.3 | 34.8 | 99.77 | 0.6 | - | - |
| 149 | 40.3 | 21.5 | 21.52 | 37.5 | 0.3 | 37.9 | 99.77 | 0.8 | - | - |
| 150 | 42.6 | 25.7 | 25.72 | 31.2 | 0.3 | 31.6 | 99.97 | 0.6 | - | - |
| 151 | 48.4 | 22.4 | 22.42 | 16.5 | 12.6 | 29.2 | 100.07 | 0.5 | 2.0/2.01 | F |
| 152 | 59.9 | 22.8 | 22.82 | 3.1 | 14.0 | 17.2 | 99.97 | 0.7 | 2.0/2.01 | F |
| 30 to 40% Amphoteric Oxides | | | | | | | | | | |
| 153 | 45.9 | 31.3 | 31.32 | 5.9 | 16.7 | 22.7 | 99.97 | 2.3 | - | - |

* = Not Fiberizable ** P = Pass, F = Failed

SUBSTITUTE SHEET

TABLE 5

FIBERS MADE WITH VARIOUS ADDITIVE CONSTITUENTS

| | ANALYSES | | | | | | 5 Hour Saline | E-119 Fire Test | |
|---|---|---|---|---|---|---|---|---|---|
| NO. | Acidic Oxides | Amphoteric Oxides | Basic Oxides | Misc. | Total | % Additive (Incl.Total) | Extraction ppm. Si | Thickness Density | 2 Hour Test |
| Fibers with $B_2O_3$ Additions | | | | | | | | | |
| 164 | 65.12 | 0.06 | 35.3 | - | 100.48 | 0.32% $B_2O_3$ | 53 | 2.0/1.94 | P |
| 165 | 64.42 | 1.20 | 34.8 | - | 100.42 | 0.52% " | 20 | 2.0/1.88 | P |
| 166 | 65.24 | 0.06 | 35.2 | - | 100.5 | 0.64% " | 43 | 2.0/1.89 | P |
| 167 | 65.32 | 0.06 | 35.2 | - | 100.58 | 0.82% " | 45 | 2.0/2.00 | P |
| 168 | 65.43 | 0.06 | 34.9 | - | 100.39 | 1.33% " | 47 | 2.0/1.95 | P |
| 169 | 65.47 | 0.06 | 34.9 | - | 100.43 | 1.37% " | 45 | 2.0/ - | P |
| 170 | 65.82 | 0.06 | 34.6 | - | 100.48 | 2.22% " | 46 | 2.0/2.02 | P |
| 171 | 68.01 | 0.06 | 32.0 | - | 100.07 | 8.41% " | 52 | 2.0/6.45 | P |
| Fibers with $P_2O_5$ addition | | | | | | | | | |
| 172 | 55.65 | 0.48 | 43.58 | 0.02 | 99.7 | 6.06% $P_2O_5$ | 71 | 2.0/1.94 | F |
| Fibers with $TiO_2$ addition | | | | | | | | | |
| 173 | 48.6 | 51.4 | - | - | 100. | 10% $TiO_2$ | 0.4 | 2.01/1.94 | P |

| NO. | Acidic Oxides | Amphoteric Oxides | Basic Oxides | Misc. | Total | % Additive (Incl. Total) | 5 Hour Saline Extraction ppm. Si | E-119 Fire Test Thickness Density | E-119 Fire Test 2 Hour Test |
|---|---|---|---|---|---|---|---|---|---|
| Fibers with $ZrO_2$ additions | | | | | | | | | |
| 174 | 63.5 | 1.10 | 35.92 | - | 100.52 | 0.21% $ZrO_2$ | 25 | 2.0/2.01 | P |
| 175 | 59.2 | 0.73 | 39.51 | - | 99.44 | 0.40% " | 48 | 2.0/2.00 | P |
| 176 | 59.5 | 0.73 | 39.52 | - | 99.75 | 0.42% " | 55 | - | - |
| 177 | 59.7 | 0.84 | 39.16 | - | 99.70 | 0.50% " | 32 | - | - |
| 178 | 60.0 | 0.90 | 38.78 | - | 99.68 | 0.54% " | 40 | - | - |
| 179 | 59.2 | 0.93 | 37.98 | - | 98.11 | 0.58% " | 46 | 2.0/2.02 | P |
| 180 | 54.3 | 1.88 | 43.12 | .01 | 99.31 | 0.58% " | 67 | 2.0/2.00 | F |
| 181 | 59.2 | 1.15 | 37.73 | - | 98.08 | 0.83% " | 57 | 2.0/2.03 | P |
| 182 | 46.85 | 2.89 | 49.98 | .02 | 99.74 | 0.84% " | 44 | 2.0/2.17 | F |
| 182a | 59.4 | 2.69 | 36.96 | .02 | 99.05 | 2.31% " | 25 | 2.0/2.00 | P |
| 183 | 59.05 | 2.95 | 38.07 | - | 100.09 | 2.65% " | 38 | 2.0/2.20 | P |
| 184 | 57.96 | 3.53 | 38.72 | - | 100.21 | 3.11% " | 25 | 2.0/2.37 | F |
| 185 | 57.80 | 3.68 | 38.14 | - | 99.62 | 3.12% " | 10 | 2.0/2.03 | F |
| 186 | 59.05 | 3.65 | 39.51 | - | 102.21 | 3.27% " | 15 | 2.1/2.11 | P |
| 187 | 56.88 | 3.62 | 40.45 | - | 100.95 | 3.30% " | 51 | - | - |
| 188 | 57.7 | 3.50 | 39.0 | - | 100.20 | 3.30% " | 13 | 2.0/2.06 | P |
| 189 | 58.19 | 3.75 | 38.65 | - | 100.59 | 3.36% " | 12 | - | - |
| 190 | 57.86 | 3.73 | 38.88 | - | 100.47 | 3.37% " | - | 2.0/2.00 | F |
| 191 | 58.6 | 4.25 | 36.22 | - | 99.07 | 3.67% " | 7 | 2.0/2.00 | P |
| 192 | 58.4 | 4.34 | 35.79 | - | 98.53 | 3.69% " | 3 | 2.0/2.00 | P |
| 193 | 58.65 | 7.87 | 35.36 | .01 | 99.89 | 4.50% " | 1.3 | 2.0/2.07 | F |

| | ANALYSES | | | | | | 5 Hour Saline Extraction | E-119 Fire Test | |
|---|---|---|---|---|---|---|---|---|---|
| Test No. | Acidic Oxides | Amphoteric Oxides | Basic Oxides | Misc. | Total | % Additive (Incl. Total) | ppm. Si | Thickness Density | 2 Hour Test |
| Fibers with $FeO_3$ additions | | | | | | | | | |
| 194 | 64.9 | 0.06 | 35.38 | - | 100.34 | 0.06% $FeO_3$ & MnO | 56 | 2.01/1.88 | P |
| 195 | 49.8 | 18.02 | 31.92 | 0.07 | 99.81 | 0.22% " " | 0.5 | - | - |
| 196 | 50.4 | 7.49 | 42.04 | 0.07 | 100.00 | 0.52% " " | 18 | - | - |
| 197 | 64.34 | 0.06 | 34.7 | - | 99.1 | 0.50% " " | 51 | 2.0/1.91 | P |
| 198 | 63.70 | 1.20 | 33.02 | - | 98.62 | 0.69% " " | 24 | 2.0/1.88 | F |
| 199 | 63.54 | 1.20 | 33.46 | - | 98.20 | 0.72% " " | 35 | 2.0/2.00 | P |
| 200 | 38.9 | 6.72 | 54.40 | 0.07 | 100.09 | 0.80% " " | 17 | - | - |
| 201 | 64.3 | 0.06 | 35.96 | - | 100.32 | 0.96% " " | 45 | 2.0/1.88 | P |
| 202 | 44.6 | 0.94 | 51.92 | - | 97.46 | 1.02% " " | 49 | - | - |
| 203 | 63.3 | 1.15 | 34.99 | - | 99.44 | 1.61% " " | 12 | 2.0/1.95 | F |
| 204 | 63.6 | 0.06 | 36.62 | - | 100.15 | 1.92% " " | 31 | 2.0/1.91 | P |
| 205 | 43.8 | 15.28 | 40.94 | 0.13 | 100.02 | 2.94% " " | 1.3 | - | - |
| 206 | 62.3 | 1.20 | 36.05 | - | 99.55 | 3.05% " " | 7 | 2.0/1.98 | F |
| 207 | 63.3 | 0.06 | 36.95 | - | 100.31 | 3.45% " " | 18 | 2.0/1.88 | F |
| 208 | 43.9 | 14.32 | 41.6 | - | 99.82 | 3.50% " " | 2 | - | - |
| 209 | 62.0 | 0.06 | 38.31 | - | 100.37 | 4.81% " " | 13 | 2.0/1.98 | F |
| 210 | 60.0 | 2.0 | 38.0 | - | 100.0 | 8.0% " " | 0.9 | 2.0/2.00 | F |
| 211 | 60.0 | - | 40.0 | - | 100.0 | 20.0% " " | 0.7 | 2.0/2.00 | F |

| | ANALYSES | | | | | | 5 Hour Saline | E-119 Fire Test | |
|---|---|---|---|---|---|---|---|---|---|
| Test No. | Acidic Oxides | Amphoteric Oxides | Basic Oxides | Misc. | Total | % Additive (Incl.Total) | Extraction ppm. Si | Thickness Density | 2 Hour Test |
| Fibers with $La_2O_3$ additions | | | | | | | | | |
| 212 | 58.1 | 0.06 | 41.47 | - | 99.63 | 0.00% $La_2O_3$ | 76 | 2.0/1.97 | F |
| 213 | 57.8 | 0.06 | 41.82 | - | 99.68 | 0.56% " | 69 | 2.0/1.97 | F |
| 214 | 57.5 | 0.06 | 41.72 | - | 99.28 | 0.72% " | 78 | 2.0/1.98 | F |
| 215 | 56.9 | 0.06 | 41.58 | - | 99.54 | 0.92% " | 70 | 2.0/1.98 | F |
| Fibers with $Cr_2O_3$ additions | | | | | | | | | |
| 216 | 62.6 | 0.51 | 36.61 | - | 99.72 | 0.09% $Cr_2O_3$ | 28 | 2.0/2.16 | P |
| Fibers with $Na_2O$ additions | | | | | | | | | |
| 217 | 64.7 | 0.06 | 35.58 | - | 100.34 | 0.28% $Na_2O$ | 45 | 2.0/1.91 | P |
| 218 | 64.5 | 0.06 | 35.68 | - | 100.21 | 0.45% " | 57 | 2.0/1.97 | P |
| 219 | 64.4 | 0.06 | 35.80 | - | 100.26 | 0.71% " | 54 | 2.0/1.97 | P |
| 220 | 63.5 | 1.20 | 35.70 | - | 100.40 | 0.87% " | 30 | 2.0/1.90 | P |
| 221 | 64.3 | 0.06 | 35.63 | - | 99.99 | 0.93% " | 51 | 2.0/1.90 | P |
| 222 | 64.2 | 0.06 | 36.11 | - | 100.37 | 1.11% " | 57 | 2.0/1.99 | P |
| 223 | 64.0 | 0.06 | 36.3 | - | 100.36 | 1.40% " | 43 | 2.0/1.99 | P |
| 224 | 63.0 | 0.06 | 37.0 | - | 100.06 | 2.60% " | 50 | 2.0/2.16 | F |
| 225 | 60.3 | 0.06 | 39.74 | - | 100.1 | 6.84% " | 70 | 2.0/1.87 | F |

| Test No. | ANALYSES: Acidic Oxides | Amphoteric Oxides | Basic Oxides | Misc. | Total | % Additive (Incl.Total) | 5 Hour Saline Extraction ppm. Si | E-119 Fire Test: Thickness Density | E-119 Fire Test: 2 Hour Test |
|---|---|---|---|---|---|---|---|---|---|
| Conventional Mineral Wool Fibers | | | | | | | | | |
| 226 | 40.0 | 9.50 | 49.97 | 0.69 | 100.16 | - | 7 | 2.0/3.50 | F |
| 227 | 39.92 | 13.99 | 45.82 | 0.74 | 100.47 | - | 1.2 | 2.0/5.23 | F |
| 228 | 38.49 | 12.24 | 49.35 | 0.61 | 100.69 | - | 0.6 | 2.0/3.42 | F |
| 229 | 41.87 | 17.10 | 41.53 | 0.64 | 101.14 | - | 1.0 | 2.0/3.86 | F |
| Refractory Fibers - (Fibers with less than 25% Basic Oxides) | | | | | | | | | |
| 231 | 31.0 | 47.52 | 21.4 | - | 99.92 | - | 2 | 2.0/2.10 | F |
| 232 | 37.1 | 59.2 | 3.3 | - | 99.6 | - | 0.6 | 2.0/5.38 | F |
| 233 | 50.0 | 40.0 | 10.0 | - | 100 | - | 0.8 | 2.0/2.00 | P |
| 234 | 54.0 | 46.0 | - | - | 100 | - | 0.3 | 2.0/2.00 | P |
| 235 | 59.62 | 25.55 | 14.23 | 0.7 | 100.11 | - | 0.3 | 2.0/2.00 | P |
| 236 | 52.1 | 46.39 | 1.13 | - | 99.62 | - | 1.0 | - | - |
| 237 | 52.0 | 46.84 | 1.07 | - | 99.91 | - | 0.4 | - | - |
| 238 | 49.8 | 49.22 | 1.02 | - | 100.04 | - | 0.3 | - | - |
| 239 | 48.6 | 50.05 | 1.00 | - | 99.65 | - | 0.4 | - | - |
| 240 | 47.8 | 51.00 | 0.98 | - | 99.78 | - | 0.3 | - | - |
| 241 | 46.2 | 53.10 | 0.93 | - | 100.23 | - | 0.4 | - | - |
| 242 | 28 | 72 | - | - | 100 | - | 0.5 | - | - |
| 243 | 64.5 | 27.4 | 8.4 | - | 100.3 | - | 0.8 | 2.0/1.85 | F |

TABLE 6

CONTINUOUS SERVICE TEMPERATURE
FOR CONSTANT $SiO_2/CaO/MgO$ RATIOS

| $SiO_2/CaO/MgO$ Ratio | 0 | 5 | 10 | 20 | 30 |
|---|---|---|---|---|---|
| | Continuous Service Temperature for max 5% shrinkage °F | | | | |
| 50/50/0 | 1480 | 1480 | 1470 | 1420 | 1550 |
| 50/40/10 | 1440 | 1430 | 1420 | 1400 | 1520 |
| 50/30/10 | 1400 | 1380 | 1370 | 1350 | 1480 |
| 60/40/0 | 1500 | 1460 | 1460 | 1460 | 1600 |
| 60/30/10 | 1430 | 1420 | 1400 | 1410 | 1520 |
| 60/20/20 | 1380 | 1370 | 1360 | 1350 | 1500 |

Reasonable modifications and variations are possible from the foregoing disclosure without departing from either the spirit or scope of the invention as defined in the claims.

CLAIMS

1. A process for decomposing a silica-containing fiber comprising the steps of:

1. providing an inorganic fiber prepared from a composition consisting essentially of:

(a) 0.06-10 wt% of a material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 35-70 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight;

2. subjecting the silica-containing fiber to a physiological saline fluid; and

3. extracting the silica at a rate of at least 5 parts per million (ppm) of silicon in 5 hours, thereby decomposing the silica-containing fiber.

2. The process of Claim 1 wherein the composition of subsection 1(a) ranges from 0.06-5 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof.

3. The process of Claim 1 wherein the composition of subsection 1(c) ranges from 0.25-50 wt% MgO.

4. The process of Claim 1 wherein the composition consists essentially of:

(a) 0.06-1.5 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-70 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

5. The process of Claim 4 wherein the composition in subsection 1(c) ranges from 0.25-50 wt% MgO.

6. The process of Claim 1 wherein the composition consists essentially of:

(a) 1.5-3 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-66 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

7. The process of Claim 1 wherein the composition of subsection 1(c) ranges from 0.25-50 wt% MgO.

8. The process of Claim 1 wherein the composition consists essentially of:

(a) 3-4 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-63 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

9. The process of Claim 8 wherein the composition of subsection 1(c) ranges from 0.25-50 wt% MgO.

10. The process of Claim 1 wherein the composition consists essentially of:

(a) 4-6 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-60 wt% $SiO_2$;

(c) 0-25 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

11. The process of Claim 10 wherein the composition of subsection 1(c) ranges from 0.25-25 wt% MgO.

12. The process of Claim 1 wherein the composition consists essentially of:

(a) 6-8 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 35-54 wt% $SiO_2$;

(c) 0-25 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

13. The process of Claim 12 wherein the composition of subsection 1(c) ranges from 0.25-25 wt% MgO.

14. The process of Claim 1 wherein the composition consists essentially of:

(a) 8-10 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 35-54 wt% $SiO_2$;

(c) 0-20 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100% by weight.

15. The process of Claim 14 wherein the composition of subsection 1(c) ranges from 0.25-20 wt% MgO.

16. The process of Claim 1 wherein the fiber has a diameter of less than 3.5 microns.

17. The process of Claim 1 wherein the silicon extraction rate is at least 20 ppm, the $Al_2O_3$ content is about 0.06-7 wt%, and the $SiO_2$ content is about 40-66 wt%.

18. The process of Claim 1 wherein the silicon extraction rate is at least about 50 ppm, the $Al_2O_3$ content is about 0.06-3 wt%, and the $SiO_2$ content is about 40-60 wt%.

19. The process of Claim 1 wherein the silicon extraction rate is at least about 50 ppm, the $Al_2O_3$ content is about 0.06-0.75 wt%, and the $SiO_2$ content is about 40-60 wt%.

20. A process of protecting a structural wall from fire comprising the steps of:

1. providing a fiber blanket having a bulk density in the range of about 1.5 to about 3 lbs. per cubic foot (pcf); wherein the fiber blanket has the ability to pass ASTM E-119 two-hour fire test; the fibers in the blanket have a diameter less than about 3.5 microns; and the fiber is an inorganic fiber prepared from a composition consisting essentially of:

(a) 0-7 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-70 wt% $SiO_2$

(c) 0-21 wt% MgO;

(d) 0-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of CaO, the total being 100% by weight; and

2. placing the blanket next to the wall, and thereby protecting the wall from fire.

21. The process of Claim 20 wherein the composition of subsection 1(a) ranges from 0.06-7 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof.

22. The process of Claim 20 wherein the composition of subsection 1(c) ranges from 0.25-21 wt% MgO.

23. The process of Claim 20 wherein the composition consists essentially of:

(a) 0.06-3.0 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58.5-70 wt% $SiO_2$;

(c) 0-21 wt% MgO;

(d) 0-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of Cao, the total being 100% by weight.

24. The process of Claim 20 wherein the composition of subsection 1(c) ranges from 0.25-21 wt% MgO.

25. The process of Claim 20 wherein the composition consists essentially of:

(a) from about 3 wt% up to and including 4 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-63 wt% $SiO_2$;

(c) 0-8 wt% MgO;

(d) 0-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of CaO, the total being 100% by weight.

26. The process of Claim 25 wherein the composition in subsection 1(c) ranges from 0.25-8 wt% MgO.

27. The process of Claim 25 wherein the composition consists essentially of:

(a) from about 4 wt% up to and including 6 wt% of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-61 wt% $SiO_2$;

(c) 0-7 wt% MgO;

(d) 0-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of Cao, the total being 100% by weight.

28. The process of Claim 25 wherein the composition of subsection 1(c) ranges from 0.25-7 wt% MgO.

29. An inorganic fiber having an average fiber diameter of less than about 3.5 microns, a silicon extraction rate greater than about 0.02 wt% Si/day in a physiological saline solution and having a composition consisting essentially of about:

(a) 0.06-5.0 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 35-70 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of Cao, the total being 100 wt%.

30. An inorganic fiber having a silicon extraction of at least about 10 ppm over a 5 hour period in physiological saline solution and having a composition consisting essentially of about:

(a) 0.06-1.5 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-70 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100 wt%.

31. An inorganic fiber according to Claim 30 having a silicon extraction of at least about 20 ppm, an average fiber diameter of less than about 3.5 microns, and having an $SiO_2$ content of about 40-66 wt%.

32. An inorganic fiber according to Claim 30 having a silicon extraction of at least about 50 ppm and having an $SiO_2$ content of about 40-60 wt% and a MgO content of about 0.25-25 wt%.

33. An inorganic fiber having a silicon extraction of at least about 10 ppm over a 5 hour period in physiological saline solutions and having a composition consisting essentially of about:

(a) 1.5-3 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-66 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100 wt%.

34. An inorganic fiber according to Claim 33 having a silicon extraction of at least about 20 ppm, an average fiber diameter of less than about 3.5 microns, and an MgO content of from about .25-50 wt%.

35. An inorganic fiber according to Claim 33 having a silicon extraction of at least about 50 ppm, an $SiO_2$ content of from about 40-54 wt%, and an MgO content of from about 0.25-18 wt%.

36. An inorganic fiber having a silicon extraction of at least about 10 ppm over a 5 hour period

in physiological saline solutions and having a composition consisting essentially of about:

(a) 3-4 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-63 wt% $SiO_2$;

(c) 0-50 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100 wt%.

37. An inorganic fiber according to Claim 36 having a silicon extraction of at least about 20 ppm, an average fiber diameter of less than about 3.5 microns, and a $SiO_2$ content from about 40-58 wt%.

38. An inorganic fiber according to Claim 37 having a silicon extraction of at least about 50 ppm and an $SiO_2$ content of from about 40-52 wt% and a MgO content of from about .25-18 wt%.

39. An inorganic fiber having a silicon extraction of at least about 10 ppm over a 5 hour time period in a physiological saline solution and having a composition consisting essentially of about:

(a) 4-6 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-59 wt% $SiO_2$;

(c) 0-46 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100 wt%.

40. An inorganic fiber according to Claim 39 having a silicon extraction of at least about 20 ppm, an average fiber diameter of less than about 3.5 microns, and an $SiO_2$ content from about 40-58 wt%.

41. An inorganic fiber having a diameter of less than about 3.5 microns and which passes the ASTM E-119 two hour fire test when processed into a fiber blanket having a bulk density in the range of about 1.5 to 3 pcf, said inorganic fiber having a composition consisting essentially of:

(a) .06-7 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-70 wt% $SiO_2$;

(c) 0-21 wt% MgO;

(d) 0.1-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of CaO, the total being 100 wt%;

wherein the amount of alumina + zirconia is less than 6 wt% and the amount of iron oxides or alumina + iron oxides is less than 2 wt%.

42. An inorganic fiber according to Claim 41 having a composition consisting essentially of about:

(a) .06-1.5 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof; and

(b) 58.5-70 wt% $SiO_2$.

43. An inorganic fiber according to Claim 42 having a silicon extraction of at least about 10 ppm over a 5 hour period in physiological saline solutions.

44. An inorganic fiber according to Claim 41 having a composition consisting essentially of about:

(a) greater than 1.5 wt% up to and including 3 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof; and

(b) 58.5-66 wt% $SiO_2$.

45. An inorganic fiber according to Claim 44 having a silicon extraction of at least about 10 ppm over a 5 hour period in a physiological saline solution.

46. An inorganic fiber according to Claim 41 having a composition consisting essentially of about:

(a) greater than 3 wt% up to and including 4 wt% material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-63 wt% $SiO_2$;

(c) .25-8 wt% MgO;

(d) .1-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of CaO, the total being 100 wt%.

47. An inorganic fiber according to Claim 46 having a silicon extraction of at least about 10 ppm over a 5 hour period in physiological saline solutions.

48. An inorganic fiber according to Claim 41 having a composition consisting essentially of about:

(a) greater than 4 wt% up to and including 6 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 58-59 wt% $SiO_2$;

(c) .25-7 wt% MgO;

(d) .1-2 wt% alkali metal oxide; and

(e) the remainder consisting essentially of CaO, the total being 100 wt%.

49. An inorganic fiber according to Claim 48 having a silicon extraction of at least about 10 ppm over a 5 hour period in physiological saline solutions.

50. An inorganic fiber having a silicon extraction of greater than about 0.02 wt% Si/day in a physiological saline solution, a continuous service temperature above about 1450°F and having a composition consisting essentially of about:

(a) .06-5 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 40-70 wt% $SiO_2$;

(c) 0-6 wt% MgO; and

(d) the remainder comprising essentially of CaO, the total being 100 wt%.

51. The fiber of Claim 50 wherein the composition of subsection (c) has an amount of 0.25-6 wt% MgO.

52. An inorganic fiber having a silicon extraction of greater than about 0.02 wt% Si/day in a physiological saline solution, having a continuous service temperature above about 1500°F and having a composition consisting essentially of about:

(a) .06-1.5 wt% of material selected from the group consisting of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides and mixtures thereof;

(b) 60-70 wt% $SiO_2$;

(c) 0-1 wt% MgO; and

(d) the remainder consisting essentially of CaO, the total being 100 wt%.

53. The fiber of Claim 52 wherein the composition of subsection (c) has an amount 0.25-1 wt% MgO.

54. An inorganic fiber according to Claims 1 or 29 made from pure oxidic raw materials.

55. An inorganic fiber according to Claim 1 or 29 or 41 in which at least a portion of the raw materials is selected from a group consisting of talc, metallurgical slags, siliceous rocks, kaolin, and mixtures thereof.

56. An inorganic fiber having a composition consisting essentially of about:

(a) 8.0-9.3 wt% $Al_2O_3$;

(b) 39-52 wt% $SiO_2$;

(c) 22-38 wt% CaO; and

(d) 7-14 wt% MgO, the total being 100 wt% and having a silica extraction in a saline solution of at least about 5 ppm over a 5 hour period.

57. An inorganic fiber composition having a composition consisting essentially of about:

(a) 49-61 wt% $SiO_2$;

(b) 10-36 wt% CaO; and

(c) 3-23 wt% MgO, the total being 100 wt% and having a $SiO_2$ extraction in a saline solution of between about 24-67 ppm over a 5 hour period.

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 4 : C03C 13/00, 13/02, 25/06 A3

(11) International Publication Number: WO 89/12032

(43) International Publication Date: 14 December 1989 (14.12.89)

(21) International Application Number: PCT/US89/02288

(22) International Filing Date: 25 May 1989 (25.05.89)

(30) Priority data: 201,513 1 June 1988 (01.06.88) US

(71) Applicant: MANVILLE SALES CORPORATION [US/US]; Manville Plaza, 5th Floor, P.O. Box 5108, Denver, CO 80217 (US).

(72) Inventors: OLDS, Leonard, Elmo ; 977 South Lake Gulch Road, Castle Rock, CO 80104 (US). KIELMEYER, William, Henry ; 3374 West Chenango Avenue, Englewood, CO 80110 (US).

(74) Agent: SCHRAMM, William, J.; Brooks & Kushman, 2000 Town Center, Suite 2000, Southfield, MI 48075 (US).

(81) Designated States: AT (European patent), AU, BE (European patent), BR, CH (European patent), DE (European patent), DK, FI, FR (European patent), GB (European patent), IT (European patent), JP, KP, KR, LU (European patent), NL (European patent), NO, SE (European patent).

**Published**

*With international search report.*
*Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.*

(88) Date of publication of the international search report: 5 April 1990 (05.04.90)

(54) Title: PROCESS FOR DECOMPOSING AN INORGANIC FIBER

(57) Abstract

Inorganic fibers which have a silicon extraction of greater than 0.02 wt% Si/day in physiological saline solutions. The fiber contains $SiO_2$, MgO, CaO, and at least one of $Al_2O_3$, $ZrO_2$, $TiO_2$, $B_2O_3$, iron oxides, or mixtures thereof. Also disclosed are inorganic fibers which have diameters of less than 3.5 microns and which pass the ASTM E-119 two hour fire test when processed into a fiber blanket having a bulk density in the range of about 1.5 to 3 pcf.

*FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT Austria
AU Australia
BB Barbados
BE Belgium
BF Burkina Fasso
BG Bulgaria
BJ Benin
BR Brazil
CA Canada
CF Central African Republic
CG Congo
CH Switzerland
CM Cameroon
DE Germany, Federal Republic of
DK Denmark
ES Spain
FI Finland
FR France
GA Gabon
GB United Kingdom
HU Hungary
IT Italy
JP Japan
KP Democratic People's Republic of Korea
KR Republic of Korea
LI Liechtenstein
LK Sri Lanka
LU Luxembourg
MC Monaco
MG Madagascar
ML Mali
MR Mauritania
MW Malawi
NL Netherlands
NO Norway
RO Romania
SD Sudan
SE Sweden
SN Senegal
SU Soviet Union
TD Chad
TG Togo
US United States of America

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US 89/02288

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

$IPC^4$: C 03 C 13/00, C 03 C 13/02, C 03 C 25/06

**II. FIELDS SEARCHED**

Minimum Documentation Searched [7]

| Classification System | Classification Symbols |
|---|---|
| $IPC^4$ | C 03 C |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

**III. DOCUMENTS CONSIDERED TO BE RELEVANT** [9]

| Category [*] | Citation of Document, [11] with indication, where appropriate, of the relevant passages [12] | Relevant to Claim No. [13] |
|---|---|---|
| X | WO, A, 87/05007 (MANVILLE CORP.) 27 August 1987 see claim 10; example III; page 5, lines 11-14 -- | 1-19 |
| A | FR, A, 1165275 (PILKINGTON BROTHERS LTD) 21 October 1958 see claim 1 -- | 1-15,17-19 |
| A | GB, A, 2083017 (NIPPON SHEET GLASS CO.) 17 March 1982 see claims 1,2; page 5, table 1, samples 9,14; page 2, lines 11-64 -- | 1-15,17-19 |
| A | GB, A, 1446910 (JAPANA INORGANIC MATERIAL CO.) 18 August 1976 see page 1, lines 22-34 -- | 1-3,6-15, 17,18 |
| A | US, A, 4366251 (RAPP) 28 December 1982 see claim 1 -- | 1,3,12-15, 17 |

./.

* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

**IV. CERTIFICATION**

| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report |
|---|---|
| 1st February 1990 | 27 FEV. 1990 |
| International Searching Authority | Signature of Authorized Officer |
| EUROPEAN PATENT OFFICE | T.K. WILLIS |

International Application No. PCT/US 89/02288

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

| Category * | Citation of Document, with indication, where appropriate, of the relevant passages | Relevant to Claim No |
|---|---|---|
| A | Chemical Abstracts, volume 81, no. 22, 27 November 1978, (Columbus, Ohio, US), see page 285, abstract 184615w, & JP, A, 7856207 (NIPPON SHEET GLASS CO., LTD) 22 May 1978 | 1,3,12-15 |
| | -- | |
| X | WO, A, 87/05007 (MANVILLE CORP.) 27 August 1987 see claims 1-10; examples I,II,III; page 5, lines 1-14; page 4, lines 13-21 | 20-29,50-53 |
| Y | | 29,41,44-49 |
| A | | 42,43 |
| | -- | |
| X | GB, A, 1446910 (JAPAN INORGANIC MATERIAL CO.) 18 August 1976 see page 1, lines 22-34; page 1, line 58 - page 2, line 32 | 50,51 |
| Y | | 29,41,44-49 |
| A | | 20-28,42,43,52,53 |
| | -- | |
| X | US, A, 2051279 (THORNDYKE) 21 March 1934 see claims 1-4; page 2, right-hand column, lines 16-49; page 2, left-hand column, lines 28-36 | 50,51 |
| Y | | 29 |
| A | | 20-28,41-49,52,53 |
| | -- | |
| A | US, A, 4366251 (RAPP) 28 December 1982 see claim 1 | 20-22,41 |
| | -- | |
| A | GB, A, 2083017 (NIPPON SHEET GLASS CO.) 17 March 1982 see claims 1,2; page 5, table 1, samples 9,14; page 2, lines 11-64 | 20-29,41-49,50-53 |
| | -- | |
| A | FR, A, 1165275 (PILKINGTON BROTHERS LTD) 21 October 1958 see claims 1,4 | 20-29,41-49,50-53 |
| | -- | |
| A | Chemical Abstracts, volume 81, no. 22, 2 December 1974, (Columbus, Ohio, US), ./.. | 20-22,41 |

International Application No. PCT/US 89/02288

**FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET**

see page 241, abstract 140076b,
& SU, A, 409981 (STATE SCIENTIFIC-RESEARCH INSTITUTE OF CONSTRUCTION MATERIALS AND PRODUCTS) 5 January 1974

--------

**V.☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]**

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1.☐ Claim numbers ..........., because they relate to subject matter not required to be searched by this Authority, namely:

2.☐ Claim numbers ..........., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3.☐ Claim numbers.........., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

**VI.☒ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]**

This International Searching Authority found multiple inventions in this international application as follows:

See Form PCT/ISA/206 dated 29th September 1989.

1.☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2.☒ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

1-19,54,55;20-28;29,50-53;41-49

3.☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4.☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☒ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

## ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. US 8902288 SA 29321

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on 21/02/90
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---|---|---|
| WO-A- 8705007 | 27-08-87 | AU-A- 6948887<br>EP-A- 0257092<br>JP-T- 63502746 | 09-09-87<br>02-03-88<br>13-10-88 |
| FR-A- 1165275 | | CH-A- 360770<br>GB-A- 810773 | |
| GB-A- 2083017 | 17-03-82 | JP-A- 57047741<br>US-A- 4443550 | 18-03-82<br>17-04-84 |
| GB-A- 1446910 | 18-08-76 | JP-A- 50014820<br>AU-A- 5892673<br>CA-A- 1022195<br>US-A- 4038089 | 17-02-75<br>06-02-75<br>06-12-77<br>26-07-77 |
| US-A- 4366251 | 28-12-82 | None | |
| WO-A- 8705007 | 27-08-87 | AU-A- 6948887<br>EP-A- 0257092<br>JP-T- 63502746 | 09-09-87<br>02-03-88<br>13-10-88 |
| GB-A- 1446910 | 18-08-76 | JP-A- 50014820<br>AU-A- 5892673<br>CA-A- 1022195<br>US-A- 4038089 | 17-02-75<br>06-02-75<br>06-12-77<br>26-07-77 |
| US-A- 2051279 | | None | |
| US-A- 4366251 | 28-12-82 | None | |
| GB-A- 2083017 | 17-03-82 | JP-A- 57047741<br>US-A- 4443550 | 18-03-82<br>17-04-84 |
| FR-A- 1165275 | | CH-A- 360770<br>GB-A- 810773 | |

EPO FORM P0479